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WRIGHT AIR DEVELOPMENT CENTER

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FOREWORD

This report was prepared by Bjorksten Research Laboratories under USAF Contract No. AF 33(038)-23319. It was administered by the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. J. I. Wittebort acting as project engineer. The work reported herein is part of the research and development program identified by RDO No. 616-13, "Optical Coatings for Aircraft Materials".

ABSTRACT

In order to dissipate precipitation electrostatic charges built up on transparent plastic aircraft canopies during flight an electrically conductive transparent coating, easily applied to formed aircraft canopies made from acrylic sheet conforming to Specification MIL-P-5425, has been developed. The coating is applied by abrading or scratching the acrylic surface slightly with a suede brush, rubbing in finely divided graphite, and spraying the surface with a protective film of a 1:5 methacrylic acid-methyl methacrylate copolymer resin.

The composite material, i. e., the acrylic and applied coating, retains essentially all the original mechanical properties, optical clarity, and light transmittance characteristics of the base material. Its surface resistance is in the range 1-10 megohms per square and is relatively insensitive to the wide variation in relative humidity, temperature, and altitude conditions that might be expected in aircraft operation. The coated acrylic is transparent to radio frequency energy as well as light, its dielectric loss factor being only slightly higher than the acrylic alone. It has sufficient permanence to the effects of sunlight, temperature variations, crazing, rain, abrasion, wind erosion, and oil absorption associated with the normal operation and maintenance of aircraft.

Three F-86 aircraft canopies, two new and one removed from service, were coated with the developed coating. The new canopies were tested in flight at Wright Air Development Center and performed satisfactorily. Thus, the coating can be applied to newly produced canopies with the least disruption in current manufacturing practices. The used canopy crazed severely when the protective coating was applied. This was caused by the strains present in the surface. Work with small methacrylate samples similarly strained revealed that the crazing could be avoided by annealing prior to spraying. Thus, it seems likely that the developed coating can be applied to canopies removed from service after an annealing pretreatment.

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PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDING GENERAL:

M. E. Sorte

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Colonel, USAF
Chief, Materials Laboratory
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DEVELOPMENT OF ELECTRICALLY CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC

INTRODUCTION

During aircraft flight through dust, snow, and ice clouds, electrostatic charges form and accumulate on the transparent plastic enclosures. Radio frequency noise resulting from the discharge of these accumulated electrostatic charges to surrounding metal structures has resulted in numerous operation failures of radio-compass and low frequency communication equipment at times when these facilities are most needed. This problem is further accentuated by the necessity for enclosing the antenna for these and other equipment within the structure of high speed aircraft. In the interests of promoting flight safety, a search was made for optically transparent coatings to be applied to acrylic plastic that would make the surfaces conductive and would, as a consequence, dissipate electrostatic charges.

From the nature of the phenomena and other engineering requirements, the developed coating must be readily applicable to formed acrylic canopies with a minimum disruption in current manufacturing practices for such canopies. In addition, the composite material consisting of acrylic and coating should retain practically all of the original mechanical properties, optical clarity, and light transmittance characteristics of the base material. The surface resistance of the coated acrylic should be 1-10 megohms per square and should be capable of conducting 100 microampere currents continuously over a foot square area without destruction. Furthermore, the surface resistivity of the composite material should be relatively insensitive to wide variations in relative humidity, temperature, and altitude conditions that might be expected in aircraft operation. Since the composite must also be transparent to radio frequency as well as light, the dielectric

loss factor should be as low as possible; i. e., preferably below 0.01 for frequencies up to 150 mc. The material must have sufficient permanence to the effects of sunlight, temperature variations, crazing, rain, abrasion, wind erosion, and oil absorption associated with the normal operation and maintenance of aircraft.

The exploratory survey of various materials which culminated in the development of an electrically conductive transparent film is described in Sections I and II, Conductive Coatings, and Protective Overcoatings, respectively. Section III describes the Test Equipment and Procedures; Section IV, Test Results; Section V, the Coating of Aircraft Canopies, involving work with new and used canopies supplied by the Air Force; and Section VI, the Recommended Procedure for the Coating Application now in use.

SECTION I

CONDUCTIVE COATINGS

The acrylic coating materials investigated included metallic deposits, resin film and graphite coatings. The application of graphite to the acrylic proved the most satisfactory electrically conducting transparent coating.

A. Metallic Coatings

A likely approach appeared to be the application to the plastic of a metallic coating with an electrical resistance of 1 - 10 megohms/square and sufficiently thin to be transparent or one that could be polished or compressed to transparency. The existence of adhesion and erosion problems with such coatings was recognized, but was subordinated to establishment of the existence of a range of suitable conductivity and transparency.

1. Silver

The deposit of a thin silver film on plastic granules prior to molding and the mirror- or silver-plating of acrylic were both tried.

a. Deposit of thin film

A thin silver coating was deposited on polystyrene granules of various sizes, prior to molding, by washing, steeping, etc. Although the films surrounding the individual granules might be expected to crack, a network of metal might remain, sufficient to insure the desired conductivity. Such a sheet could then be applied as a transparent surface coating with the fine metal reticle remaining. However, the resultant molded product was of insufficient conductivity or insufficient transparency.

b. Mirror-plating

From the standpoint of conductivity this procedure appeared simpler, although the problems of adhesion and erosion would be more difficult.

In using the Brashear process, the material to be coated was pretreated with solvents or by surface blasting or tumblihg to improve adhesion, with wetting agents to improve the uniformity of coating, and with stannous chloride for the customary sensitization. Various organic reducers were also tried, including the hydroquinone type, hydrazine, and the tartrates. None of these methods produced a coating adequate in both transparency and conductivity.

The Rochelle salts plating procedure described in the Handbook of Physics and Chemistry, 31st Edition, pp. 2590-1, was adapted as follows:

i. Cleaning

Methacrylate samples to be plated were immersed and swabbed in isopropyl or ethyl alcohol, rinsed in distilled water, immersed with agitation in 5% NaOH solution for 5 minutes, rinsed in distilled water, immersed with agitation in 15% nitric acid for 3 minutes and finally rinsed well in distilled water.

This procedure was necessary to insure even plating, although cleaning with alcohol alone occasionally yielded good results.

ii. Wetting and Sensitizing

To promote the subsequent wetting of the surfaces, the samples were dipped briefly into a 2% solution of a wetting agent such as Aerosol OT. The samples were then sensitized by a 3-minute immersion in stannous chloride-hydrochloric acid solution (10 grams of stannous chloride and 40 ml. concentrated hydrochloric acid plus water to make 1000 ml.). Careful washing was necessary after sensitization to remove any excess stannous chloride.

iii. Preparation of Plating Solutions

Plating Solution

Dissolve 5 grams of silver nitrate in 300 ml. of distilled water.

Add dilute aqua ammonia until the precipitate formed is nearly, but not entirely, dissolved.

Filter and add sufficient distilled water to the filtrate to make 500 ml.

Reducing Solution

Dissolve 1 gram of silver nitrate in a small quantity of water and add to about 500 ml. of boiling water.

Add 0.83 grams of Rochelle salts in solution to the boiling solution.

Continue to boil the solution for about 30 minutes until a gray powder settles out.

Filter while the solution is hot. Allow it to cool and add sufficient distilled water to make 500 ml.

iv. Plating Procedure

Equal volumes of the above solutions were mixed just before use and poured over samples resting in an enameled tray. The solution was agitated for from 20 seconds to 2 minutes according to the thickness of mirror desired. The samples were then removed and thoroughly rinsed in water.

v. Results

Some of the first samples plated as above were quite encouraging. For example, sample ED-46 had a resistance of 25-35 ohms/square and a light transmittance of 19%. Since the desired resistance was 10^5 times this value, it seemed likely that the thickness of the silver plating could be reduced considerably to increase the light transmittance satisfactorily before too high a resistance was encountered. However, the resistance of the silver films increased more rapidly than the light transmittance, exceeding the maximum resistance limit (10 megohms/square) long before minimum transmittance limit (80%) was approached. This relationship is shown graphically in Figure 1, which is drawn from the data given in Table No. 1.

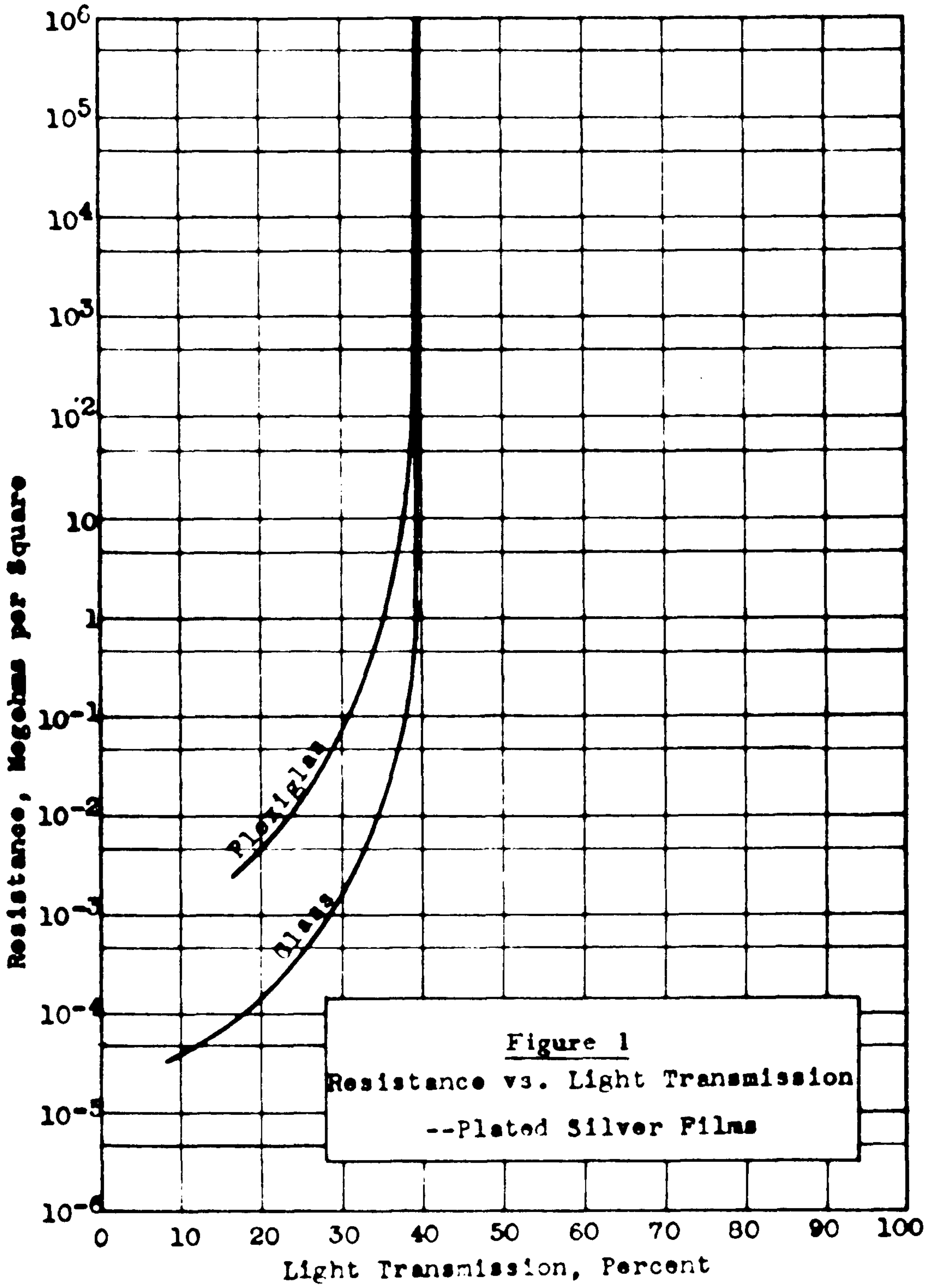

Figure 1
Resistance vs. Light Transmission
--Plated Silver Films

TABLE NO. 1

DATA ON PLATED SILVER COATINGS

Sample No.	Substrate	Plating Time[a] (seconds)	Resistance (megohms/ square)	Light Transmittance (percent)
ED-11-30	Plexiglas II	30	∞ [b]	84
ED-11-60	Plexiglas II	60	∞	69
ED-6-7	Plexiglas II	60	18[c]	38[d]
ED-12-1	Plexiglas II	60	0.1	31[d]
ED-20-30	Glass	30	∞	54[d]
ED-20-40	Glass	40	∞	48[d]
ED-20-50	Glass	50	∞	45[d]
ED-20-60	Glass	60	∞	39[d]
ED-18-1	Glass	60	3.4×10^{-3} [c]	30[d]
ED-18-2	Glass	120	6.8×10^{-5} [c]	15[d]

(a) With dilute (1:4) Rochelle salt plating mixture.

(b) Resistance probably over 100,000 megohms per square.

(c) Average values on uneven samples.

(d) Values below range of haze meter obtained by using Weston Master photographic exposure meter.

The plated silver films were unsatisfactory in other respects. They were found to be quite uneven, despite their apparent evenness to the naked eye. Resistance values obtained between points 1/4" apart varied from 0.2 megohms to infinite megohms/square over an area of 3 square inches.

The films as deposited were also non-adherent, being removed by a light rubbing with the finger. Films were made to adhere by spraying the methacrylate sample beforehand with an adhesive. However, the adhesive increased the resistance of the resultant film due, at least in part, to microscopic cracking of the adhesive layer.

2. Deposits of Other Metals

Iron powder (GAF Carbonyl E, General Aniline and Film Corp.) and aluminum powder were separately rubbed on pre-scratched methacrylate sheets. A considerable amount of the powder was deposited in the scratches in both cases, as was observed under the microscope.

None of these samples showed any conductance until the deposited coating became so thick that it was practically opaque.

3. Metallic Compounds as Coatings

a. Titanium Compounds

Titanate coatings, which are quite adherent and transparent, can be made conductive by reduction to titanium by hydrogen at an elevated temperature. Attempts were made to promote this reduction at a temperature tolerated by plastic sheets by using stronger reducing agents such as hydrazine and nascent hydrogen. All results by this method were negative.

Titanium dioxide films were prepared by coating methacrylate samples with 5% solutions of tetra isopropyl titanate and tetrabutyl titanate in heptane. After drying in air, the coatings produced non-adherent films. The film from tetra isopropyl titanate was white and translucent with a surface resistance of 35,000 megohms/square, and from the tetrabutyl titanate, the film was transparent but uneven and with a resistance of over 100,000 megohms/square.

b. Molybdenum Disulfide

This compound has a plate-like crystalline form similar to graphite and is electrically conductive. Its use as a conductive coating comparable to those obtained with graphite was investigated.

A finely powdered form of this disulfide (Climax Molybdenum Co. Molysulf Grade 2) was rubbed onto a pre-scratched methacrylate plate. No conductance was observed until the coating was so thick as to be opaque.

c. Phlogopite Mica

This material was suggested as a transparent material of the same physical structure as graphite and as having electrical conductance in the range required for this project. It was found, however, to have infinite surface resistance and was therefore unsatisfactory.

B. Resin Film Coatings

1. "Markite" Conducting Plastic

The Markite Corporation reported that this type of conducting plastic is not available in solution form because of inherent properties and thus could not be considered as a coating material.

2. Chloride-containing Polymers

The thermal decomposition of polymers containing chlorides might produce enough ionized hydrogen chloride for conductivity in the desired range.

Solutions of various Vinylites (containing vinyl chloride), Pliolite, and Saran were made and coated on glass. Heating at temperatures up to 250°C. did not produce conducting films.

3. Plasticized Nylon Films

Several films were prepared using Nylon Type 8 with various other ingredients. A 60:40 mixture of Nylon and diethylacetamide was milled and then pressed for several minutes at 110°C. The surface resistance was only 2 megohms/square shortly after being pressed, but increased to 4,000 megohms/square in one hour.

Films with various ratios of Nylon Type 8, plasticized with dimethylformamide containing 4% of barium nitrate, showed low surface resistance shortly after preparation, but their resistances increased to as much as 500 megohms/square within 18 hours. A portion of one of these was dissolved in methanol and a new film formed on a glass plate by evaporation of the solvent. The resistance of this film was about 10 megohms/square after drying, but increased to 4,000 megohms/square after 16 hours.

4. Polyacrylic-polyamine Copolymers

Aqueous solutions of polymethacrylic acid were mixed with varying proportions of ethylene diamine and the mixtures applied to methacrylate surfaces. When these were heated under an infrared lamp, tough, adherent films were obtained. These were unacceptable as conductive coatings, however, because of their sensitivity to the ambient relative humidity - the electrical resistance varying from 200 to 3,000 megohms/square with fluctuations in laboratory humidity over a two week period.

Polymethacrylic acid was also combined with triethylene tetramine and hexamethylene diamine as above. The products obtained with the latter compound yielded the most promising of all coatings containing amines. Coatings containing various concentrations of hexamethylene diamine showed surface resistances of 30-40 megohms/square during two weeks exposure in the laboratory. These results were promising enough to indicate the advisability of further work with these materials. Because of more promising immediate results with graphite coatings, the laboratory work was concentrated upon their perfection.

5. Polyvinyl Chloride

Films prepared by pressing polyvinyl chloride plasticized with tricresyl phosphate (60:40) between glass plates had surface resistances of 10,000 megohms/square.

6. Urethan Resin

A urethan film was prepared by mixing sodium tartrate dissolved in ethylene glycol and methyl ethyl ketone with metatolylene diisocyanate and pouring the mixture on a glass plate. When moist, this film had a surface resistance of 2 megohms/square. When dry, its resistance was infinite.

7. Metallic Salts in Resins

Films of methylmethacrylate-methacrylic acid copolymer containing stannous or sodium chlorides were prepared by mixing the copolymer solution and the salt and pouring onto a glass plate. The dry films were quite hard and showed infinite resistance. The addition of small amounts of glycerine caused the films to remain moist and show resistances of 1 to 10 megohms/square, but then the films did not adhere to methacrylate surfaces.

C. Graphite Coatings

The most successful coatings produced in this investigation were prepared by rubbing finely divided graphite into microscopic scratches in polymethacrylate surfaces. By this procedure, coatings were obtained with resistances and transparencies well within the prescribed ranges.

The recommended procedure which was developed requires scratching the methacrylate surfaces, application of the graphite, polishing in the graphite, and finally the application of a protective overcoating.

1. Scratching Methacrylate Surfaces

a. New Surfaces

During the preliminary work with graphite, there was some uncertainty as to the actual mechanism of conduction. A treated surface showed three fairly distinct regions as regards the distribution of the graphite. Under a magnification of 400X, it was possible to observe: (1) a network of fine scratches filled with graphite; (2) scattered particles of graphite, non-uniform both in size and in distribution over the surface; and (3) a grayish homogeneous film.

All three phenomena were found on all treated samples, but in varying extents. At first it was thought that the homogeneous film was mainly responsible for the conductance, since some specimens with very few scratches were found to be adequately conductive. But further work showed that the chief avenues of conduction were the lines and scratches filled with graphite tightly compressed by vigorous rubbing.

The effectiveness of these lines of graphite was shown by preparing samples with unidirectional scratches. When graphite was applied to such a surface and rubbed into the scratches, the surface resistance along the scratches was always much less than that measured across the scratches, Such samples showed a resistance of a few megohms/square in the direction parallel to the scratches and infinite resistance in the direction at right angles to the scratches. Thus, in Figure 2, a surface abraded in one direction is shown. The surface resistance was 2.9 megohms/square in that direction and was infinite in the perpendicular direction. Figure 3 illustrates a surface with abrasions in perpendicular directions with a surface resistance of 7-8 megohms/square.

Figure 2. Negative Photomicrograph of Coated Acrylic Surface with Unidirectional Scratches. These have been filled with graphite and sprayed with the copolymer overcoating.

Figure 3. Negative Photomicrograph of Coated Acrylic Surface. The surface has been abraded in perpendicular directions and treated with graphite and the copolymer.

Meanwhile, closer examination of the homogeneous film mentioned above showed this to be a network of very fine graphite-filled scratches. It was, therefore, decided that the first step in applying a graphite conductive coating would be the abrasion of the surface to produce fine, interconnecting scratches.

Several methods were used for producing such scratches or lines on methacrylate surfaces. Just rubbing the surface with a cloth, as in applying and rubbing in the graphite, finally produced a network of scratches which become filled with graphite and give adequate conduction. It seemed better, however, to produce the scratches by some faster and more controlled method.

Silicon carbide (Carborundum) of various grit sizes (100 to 600 mesh) was mixed with dry starch in varying proportions (5 to 15% of abrasive) and the mixtures rubbed on methacrylate samples with a cloth. The pattern of scratches so produced depended upon the coarseness and proportion of abrasive, and the amount, direction and pressure of rubbing. This procedure produced an excellent base for the graphite coating, but it tended to mar the surface and thereby reduce its optical clarity. This method was also difficult to control in obtaining a very light abrasion of the specimen.

Attempts were made to use both rotary and belt-type polishing or sanding devices for this operation. The polishing wheel or belt was covered with cloth and used for rubbing on the starch-abrasive mixtures mentioned above. The resulting abrasion was rather uneven despite all possible care in manipulation, so that the above mechanical scratching did not seem to offer any advantage over the manual operation.

Very fine sandpaper and crocus cloth were used for making scratches on methacrylate. Such scratches were too deep and too numerous, greatly reducing the optical clarity.

One method of standardizing the scratching operation seemed to lie in the use of sharp points or blades evenly pressed against the surface to be coated. A large number of methacrylate sheets were treated in this manner and a method developed which yielded a coated surface with high light transmittance value and satisfactory conductance. After trying phonograph needles and pointed gem stones set into steel rods, attention was centered on razor blades as ideal for this grooving operation. The blades were supported on a rod in a small rolling frame which was drawn across the sample. The depth and number of grooves were easily varied to give some control over the resultant conductance and clarity of the finished specimen.

The standardized abrading device which proved most satisfactory for producing scratches on new methacrylate surfaces was a small brass-bristled brush, commonly called a "suede brush". The numerous fine wires of this brush produced innumerable fine scratches of the right depth when it was rubbed with moderate pressure over the surface. One stroke of the brush over an area was ample for coverage but a second stroke at right angles was often made to insure adequate conductance in all directions. A small brush of this type is shown in use on a small plate in Figure 4. For treatment of larger surfaces such as aircraft canopies, larger brushes are recommended.

b. Surfaces of Used Aircraft Canopies

After being in service for some time, canopies become scratched to some extent over most of their surfaces with severe scratching in some spots. Such surfaces must be cleaned and the deep scratches removed before application of a graphite conductive coating. A thorough polishing with an abrasive-containing polishing compound (such as Du Pont No. 7) removed the blemishes from such a surface and the scratches necessary for rubbing-in of the graphite. Special attention must be given to the spots with severe scratching,and any waxy or oily residue from the polishing compound or from finger marks must be removed to avoid smudging of the graphite.

2. Application of Graphite

Initially, graphite was spread over the surface to be coated by light rubbing with a piece of soft paper or cloth. Although there have been some modifications and improvements in the details of this operation, the recommended procedure is essentially the same.

Mechanical application with rotary buffers was unsatisfactory and only fairly satisfactory with a bench-type sander specially adapted for this operation. The sanding belt was replaced by a belt covered with soft cloth and was operated at a speed of about 400 ft. per minute. The cloth was saturated with graphite and the scratched surface held against it. Graphite was deposited on the surface, especially in the scratches when these were at right angles to the travel of the belt. The conductivity of the coating depended upon the time the sample was treated and the pressure applied during the "buffing". This operation is a combination of applying the graphite and polishing it in, as described below. It is quite satisfactory for small, flat samples but could not be readily adapted to large or curved pieces.

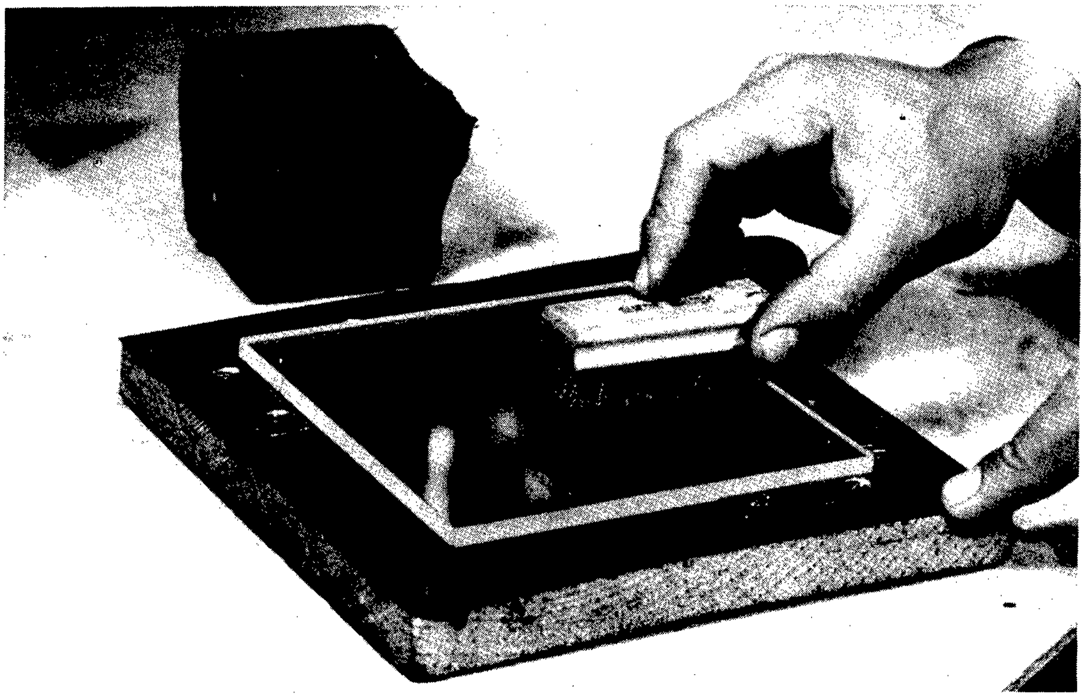

Figure 4. Abrasion of Methacrylate Surface with Suede Brush.

The best method developed for applying graphite was the manual use of a soft, flannel-type cloth saturated with graphite. This cloth was fastened over a sponge-rubber pad fixed on a wood block of convenient size. The cloth was saturated with graphite (Dixon Airspun Graphite, Type 200-10) by spreading it on and rubbing it in with a spatula. The surface being treated was rubbed over thoroughly with this applicator using longitudinal, vertical and rotary motions to insure the filling of all scratches on the surface.

3. Polishing in the Graphite

For adequate conductance of the coating with a minimum of graphite, the graphite must be compressed into the surface scratches as much as possible without being rubbed out again. Dependent upon whether graphite is to be removed or additional graphite to be applied, a clean or graphited cloth similar to the applicator pad above was used. Considerable pressure was applied to wedge the graphite particles firmly into the scratches. In treating a large surface, such as an aircraft canopy, small areas where electrical resistances were above or below the desired range were rubbed further, using a clean or a graphited cloth as required.

By means of this procedure, conductive coatings with electrical resistances between 1 and 10 megohms/square, a light transmittance of 90% or better, and a haze value of less than 2% were produced consistently.

Variations in the use of graphite were attempted in the search for the best possible material and method of application. Acetylene black was substituted for graphite in the simple rubbing-in process, but did not cling to the surface nor become packed into the scratches as did graphite. There was no measurable conductance over such a treated surface. Acetylene black was also incorporated with plasticized polyvinylbutyral and Uformite F-240-N and the mixtures suspended in methanol. Lines of these suspensions were put on methacrylate surfaces and allowed to dry. Resistances of 0.1 to 5 megohms/square were measured on these surfaces, but the light transmittance was greatly reduced by this treatment.

Powdered metals, zinc and silver, were mixed with graphite and rubbed on scratched methacrylate surfaces. The metals alone did not adhere well to methacrylate but when graphite was added, in amounts as low as one part in ten, there was adequate adhesion of the mixture. These samples showed satisfactory resistances if the proper amount of the mixture was applied.

These metal-graphite coatings appeared to be more transparent than graphite coatings with the same conductivity, but measurements revealed that percentages of light transmittance were about the same for the two types of samples. The apparent superiority of the zinc- or silver-containing type was probably due to the lighter color of the metal. One marked disadvantage of using metal in the coating is that the metal particles, when packed into the fine scratches, reflected enough light to make the pattern of scratches stand out noticeably and thus cause added optical interference.

The recommended graphite coating was not resistant to weathering, being removed merely by washing with water. The development of an overcoating to protect the graphite coating without affecting its desirable characteristics is described in Section II.

SECTION II

PROTECTIVE OVER-COATING

To preclude removal by water and/or other atmospheric conditions of the graphite conductive coating from the methacrylate surface to which it had been applied, a protective coating was needed. This over-coating must not only resist weathering, but should be transparent, adherent to methacrylate, easy to apply, and should not adversely affect the conductivity of the graphite.

The first composition tried for such an over coating was a 1:5 mixture of methacrylic acid and methyl methacrylate copolymerized in 10 parts of Cellosolve acetate by refluxing with 0.5% benzoyl peroxide for one hour. The viscous liquid obtained was diluted with more Cellosolve acetate; 1:1 for roll coatings and 1:8 for spraying.

When this copolymer coating was applied to a graphited surface, the electrical resistance usually increased by 1 to 6 megohms/square. The light transmittance sometimes increased slightly, but more often it decreased considerably and considerable haze was produced if proper care was not used in the application of the copolymer. The over-coating had a thickness of 0.001" to 0.002" when rolled on and of less than 0.001" when sprayed on.

The ability of the methacrylic acid-methyl methacrylate copolymer to cover the graphite coating and yet to permit conductance over the treated surface seems to lie in the acid component. Apparently the acid supplies mobile ions which permit conductance through the lacquer to the graphite. For example, one sample with the graphite coating had a resistance of 0.6-0.9 megohm/square before the application of the overcoating. After overcoating with the copolymer in the ratio of 1:5 methacrylic acid to methacrylate, the resistance increased to 25 megohms. Using the 1:9 ratio of methacrylic acid to methacrylate increased the resistance to only 3-6 megohms.

Modifications of this formulation were prepared to improve the hardness, adhesion, weather resistance, sprayability, etc. Results with these modifications are presented in Table No. 2.

TABLE NO. 2

GRAPHITE COATINGS AND COPOLYMER OVERCOATINGS

Sample No. and Size	Treatment: Conductive Coating	Treatment: Polyelectrolyte Overcoating	Treatment: Method of Over-coating	Surface Resistance (Megohms/Square) Before Overcoating	Surface Resistance (Megohms/Square) After Overcoating	% Light Trans-mittance	% Haze
0-500 3" x 3"	Graphite rubbed on	1:5 copolymer ES-1 in cy-clohexanol 1:1	Rolled	0.1-7	7-660		
0-501 3" x 3"	Prescratched with emery 302-1/2 graphite rubbed on.	1:5 copolymer ES-1 in cy-clohexanol 1:1	Rolled	1.3-2.7	4		
0-504 3" x 3"	Phosphen 5 (Dow)-used as a lubri-cant and wet-ting agent - rubbed on with graphite	1:5 copolymer ES-2 in cy-clohexanol 1:1	Rolled	0.7-2.7	4-9.3	90.1	2.7
0-507 6" x 6"	Phosphen 5 and emery 302-1/2 mixed and rubbed on with graphite	1:5 copolymer ES-2 in Cel-losolve acetate 1:1	Rolled	4-20	9.3-300		
0-508 3" x 3"	Prescratched with silicon carbide (320 mesh), Graphite rubbed on.	1:5 copolymer ES-2 in cy-clohexanol 1:1	Rolled	2-13	30-40	85	4.4

TABLE NO. 2 (Cont.)

GRAPHITE COATINGS AND COPOLYMER OVERCOATINGS

Sample No. and Size	Treatment			Surface Resistance (Megohms/Square) Before-After Overcoating		% Light Transmittance	% Haze
	Conductive Coating	Polyelectrolyte Overcoating	Method of Over-coating	Before	After		
0-510 6" x 6"	Prescratched with silicon carbide (320 mesh) and starch - graphite (1:1) rubbed on.	1:5 copolymer ES-2 in cy-clohexanol 1:1	Rolled	1-2.7	2.7-4		
0-513 6" x 6"	Prescratched with 302-1/2 emery, Phosphen 5 rubbed on; then graph-ite.	1:5 copolymer ES-2 in cy-clohexanone 1:1	Rolled	1.0-7	4.5-6		
0-519 3" x 3"	Prescratched with silicon carbide (320 mesh) and starch - graphite (1:1) rubbed on.	1:5 copolymer ES-1 in cy-clohexanol 1:1	Rolled	2.7-10.5	7-20		

TABLE NO. 2 (Cont.)

GRAPHITE COATINGS AND COPOLYMER OVERCOATINGS

Sample No. and Size	Treatment: Conductive Coating	Treatment: Polyelectrolyte Overcoating	Treatment: Method of Over-coating	Surface Resistance (Megohms/Square) Before Overcoating	Surface Resistance (Megohms/Square) After Overcoating	% Light Trans-mittance	% Haze
0-521 3" x 3"	Prescratched with silicon carbide (320 mesh) and 1:1 starch-graphite rubbed on.	1 part ES-1 2 parts butyl acetate 1 part n-butanol	Rolled	4-20	13-53		
0-524 3" x 3"	Same as above	1 part ES-1 2 parts methyl isobutyl ketone	Rolled	4-5.3	5.5-8	90.2	2.7
0-526 3" x 3"	Same as above (scratched in one direction)	1 part ES-1 3 parts methyl isobutyl ketone	Rolled	2.7-660	2.9-∞		
0-528 3" x 3"	Prescratched with silicon carbide (320 mesh) and 1:1 starch-graphite rubbed on.	1 part ES-1 8 parts methyl isobutyl ketone	Sprayed	4.2	6.6-8	87.6	4.2

TABLE NO. 2 (Cont.)

GRAPHITE COATINGS AND COPOLYMER OVERCOATINGS

Sample No. and Size	Treatment: Conductive Coating	Treatment: Polyelectrolyte Overcoating	Treatment: Method of Over-coating	Surface Resistance (Megohms/ Square) Before Overcoating	Surface Resistance (Megohms/ Square) After Overcoating	% Light Trans-mittance	% Haze
0-530 6" x 6"	Prescratched with silicon carbide (320 mesh) and 1:1 starch - graphite rubbed on.	1 part ES-1 2 parts butyl acetate 1 part n-butanol	Sprayed	1.4	2.0-4.5	73.7	5.1
0-531 6" x 6"	Same as above	Same as above	Sprayed	2.2	3.7-4.5	79	4
0-538 24"x24"	Same as above	Same as above	Sprayed	1.4	1.4		
0-539 6" x 6"	Same as above	1 part ES-5 1 part ethylene dichloride	Sprayed	1.4	5-6		
0-540 6" x 6"	Same as above	1 part ES-5 2 parts methyl-ethyl ketone	Sprayed	1.4	1.5	80	5.1
0-542 6" x 6"	Same as above	1 part ES-1 4 parts ethyl lactate 8 parts methyl ethyl ketone 1% Aerosol OT	Sprayed	1.5	1.4		
0-550 6" x 6"	Same as above	1 part ES-1 3 parts methyl isobutyl ketone	Sprayed	1.9-3	6-7		

Copolymer ES-1 was prepared by refluxing methacrylic acid (0.2 mole) and methyl methacrylate monomer (1 mole) in cyclohexanone (2 moles) with benzoyl peroxide catalyst (0.2% of total weight). The mixture was refluxed (137-138°C.) for one hour and then abruptly cooled in an ice-water bath. Copolymers ES-4 and ES-8 were made in exactly the same manner.

Copolymer ES-2 was prepared in a similar manner using ethylene glycol monoethyl ether acetate as the solvent instead of cyclohexanone.

Copolymer ES-5 was prepared in the same manner as ES-1 except that the ratio of methacrylic acid to methyl methacrylate was 1:7.

Because of the viscosity of these copolymers, they were dissolved in various solvents and rolled or sprayed on graphite-coated specimens. The solvents used were cyclohexanol, cyclohexanone, butanol-butyl acetate, methyl ethyl ketone, methyl isobutyl ketone, ethylene dichloride, and an ethyl lactate-methyl ethyl ketone-Aerosol OT mixture.

Satisfactory rolled coating formulations included:

(1)	Copolymer ES-1,	1 part
	Butyl Acetate,	1 part
	N-butanol	1 part
(2)	Copolymer ES-1,	1 part
	Methyl Isobutyl Ketone,	2 parts
(3)	Copolymer ES-1,	1 part
	Methyl Isobutyl Ketone,	3 parts

Satisfactory spray coating formulations included:

(1)	Copolymer ES-1,	1 part
	Methyl Isobutyl Ketone,	8 parts
(2)	Copolymer ES-1,	1 part
	Butyl Acetate,	2 parts
	N-butanol	1 part
(3)	Copolymer ES-1,	1 part
	Methyl Isobutyl Ketone,	3 parts

(4)	Copolymer ES-5,	1 part
	Ethylene Dichloride,	1 part
(5)	Copolymer ES-5,	1 part
	Methyl Ethyl Ketone	2 parts
(6)	Copolymer ES-1,	1 part
	Ethyl Lactate	4 parts
	Methyl Ethyl Ketone,	8 parts
	1% Aerosol OT 100%	

Spraying was the preferred method of application and the best formulation was (2) above with methyl ethyl ketone in place of n-butanol, so that the recommended formulation which was used in all subsequent work was:

Copolymer ES-1,	1 part
Butyl Acetate,	2 parts
Methyl Ethyl Ketone,	1 part

Methyl ethyl ketone seems to tolerate more of the copolymer than does n-butanol so that there is less possibility of precipitating the copolymer.

Triallyl cyanurate, which forms clear, hard polymers was used to form a copolymer with methacrylic acid and methyl methacrylate. A mixture of these three, containing 0.1 mole, 0.2 mole, and 1 mole respectively, was dissolved in 2 moles of cyclohexanone with 0.2% benzoyl peroxide and refluxed for one hour. The copolymer thus formed (ES-6) was harder than the 1:5 copolymers without the cyanurate. But the use of this overcoating on graphite-coated specimens caused a greater increase in resistance (5 to 15 megohms) than was caused by the copolymers without cyanurate.

A copolymer (ES-11) of methacrylic acid and cyclohexyl methacrylate (1 to 5 molar) was prepared similarly to ES-1. This substitution of a component with longer side chains into the copolymer seemed to increase the hardness of the product but further tests showed that this was not accomplished.

Attempts to copolymerize methacrylic acid and vinyl benzoate were unsuccessful as were attempts to use other vinyl lacquers.

Coatings with greater hardness than shown by some of the copolymer coatings were prepared with General Electric R-108 Intermediate. The following formulation, based on recommendation by the manufacturer, was used for the initial preparation.

Polyvinylbutyral, 5 parts, was dissolved in 50 parts of a solvent mixture of s-butanol and diacetone alcohol (85:15). This solution was added to R-108 Intermediate (45 parts) containing phosphoric acid (0.75 part in 10% alcoholic solution) as a catalyst. For a rolled coating, this mixture was diluted to 35% solids by adding solvent mixture. For spraying, it was further reduced to 17.5% solids.

The overcoating was hardened by heating at 145°C. for one-half hour, or at 100°C. for 3 hours, or 90°C. for 4 hours. Heating at 70°C. for 8 hours was not sufficient to cause adequate hardening.

Specimens coated with this lacquer gave satisfactory tests for resistance and light transmittance. The haze value was excessive, but was reduced to an acceptable value by the addition of butyl acetate (20%) to the solvent mixture during formulation.

The coating was not removed nor visibly scratched by the prescribed polishing operation, which removes some acrylic copolymer coatings. However, specimens coated with this material were severely discolored when exposed to accelerated weathering. Consequently, this material was not used further.

SECTION III

TEST EQUIPMENT AND PROCEDURES

The samples prepared in the course of development were evaluated in terms of the following properties:

A. Surface Resistance
B. Haze and Light Transmittance
C. Resistance to Accelerated Weathering
D. Resistance to Polishing
E. Adhesion of the Coating
F. Mar Resistance and Hardness
G. Tensile, Flexural, and Impact Strengths
H. Dielectric Loss Factor

A. Surface Resistance

Because it took 5 minutes or longer to measure the electrical resistance of coated surfaces with the apparatus described in Federal Specification L-P-406a, Method 4041, the equipment in Figure 5 was used.

The sample holder at the left has adjustable electrodes of thin metallic aluminum strips mounted on sponge rubber. These are 5 inches long and may be spaced from 1 to 4 inches apart to accommodate various sizes of samples. The sponge rubber permits better contact with curved or irregular surfaces. The small electrodes on the right were used for measuring resistances on smaller samples or on coated canopies where it was important to measure small areas. These metal strips are 3/4" apart, with a flat portion 5/8" long which makes contact with the surface being tested.

The electrodes were connected to the Hickok Meter, Model 209A, as a substitute for the galvanometer, shunts, etc., described in Federal Specification L-P-406a. When set for the proper function and range, the readings on the resistance scale of this instrument were always found to agree with known resistances inserted across the electrodes.

During testing, the coated surface was pressed firmly against the electrodes, or vice versa. On small samples, two to four readings were taken in different positions to give an average for the sample. In testing a coated canopy, measurements were taken at a large number of points to give an average of over-all resistance and to detect any spots with resistances outside the allowable limits.

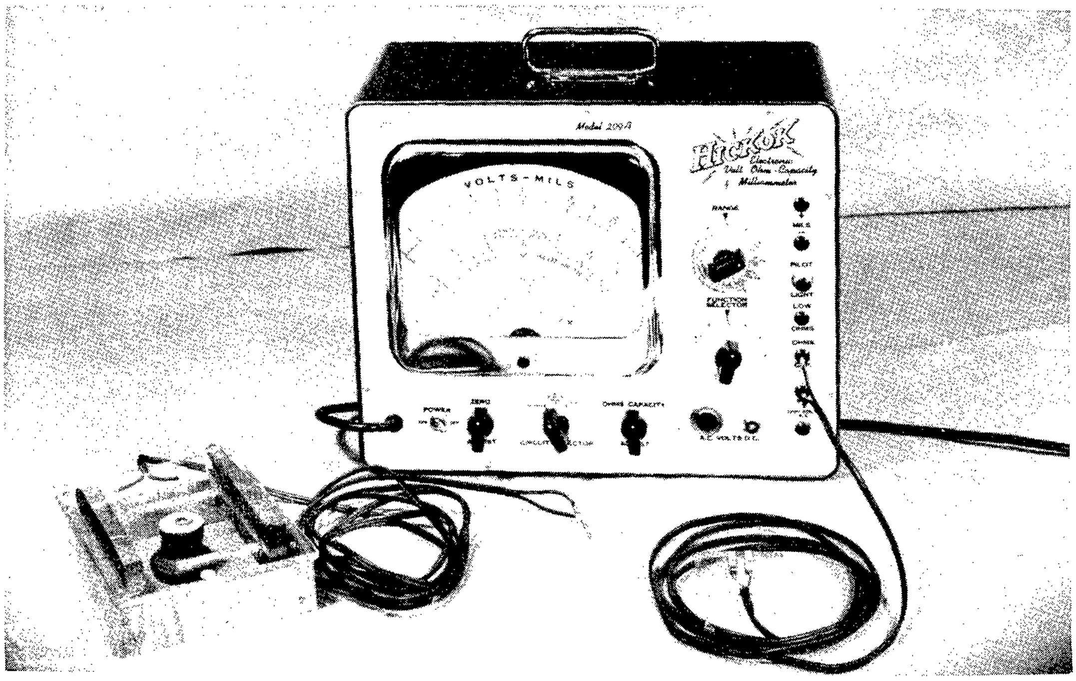

Figure 5. Equipment for Measuring Surface Resistance.

B. Haze and Light Transmittance

1. On Small Samples

The haze value and light transmittance were measured with an instrument built according to Method 3021 of Federal Specification L-P-406a. (Figures 6 and 7).

The optical system includes a 250 watt lamp, reflector and condensing lens at one end, and a Weston Photronic photoelectric cell at the other end. The cell and the aperture of the condensing lens are enclosed in a box with dull black finish inside. The two apertures are 18 inches apart..

The electrical measuring system was built according to one used by Campbell and Freeth (J. Sci. Instr., 11 (4): 125, 1934) and by Fogle (Trans. Elec. Chem. Soc., Vol. 66, 1934).

The circuit was derived from one contained in the general instructions for using the Weston Photronic Cell, Model 594 - Type 3. This circuit indicates a method of obtaining the equivalent of a zero external circuit resistance. The current from the aiding battery compensates for the Photronic Cell e.m.f. so that, when the galvanometer is balanced, the meter indicates the cell current for zero potential across the cell. A Simpson Model 260 volt-ohm-milliammeter, with a sensitivity of 20,000 ohms per volt, is being used in place of a galvanometer. On the 100 microamp range, its internal resistance is 1000 ohms.

Haze is calculated as follows:

$$\% \text{ Haze} = \frac{T - T_r}{T} \times 100 = \frac{T_d}{T} \times 100$$

where T = total light transmitted

T_r = amount of light transmitted rectilinearly

T_d = amount of light not transmitted rectilinearly

$$\frac{T - T_r}{T} = \frac{I - I_r}{I}$$

where I and I_r are the currents read directly from the meter as the values for two specified positions of the sample.

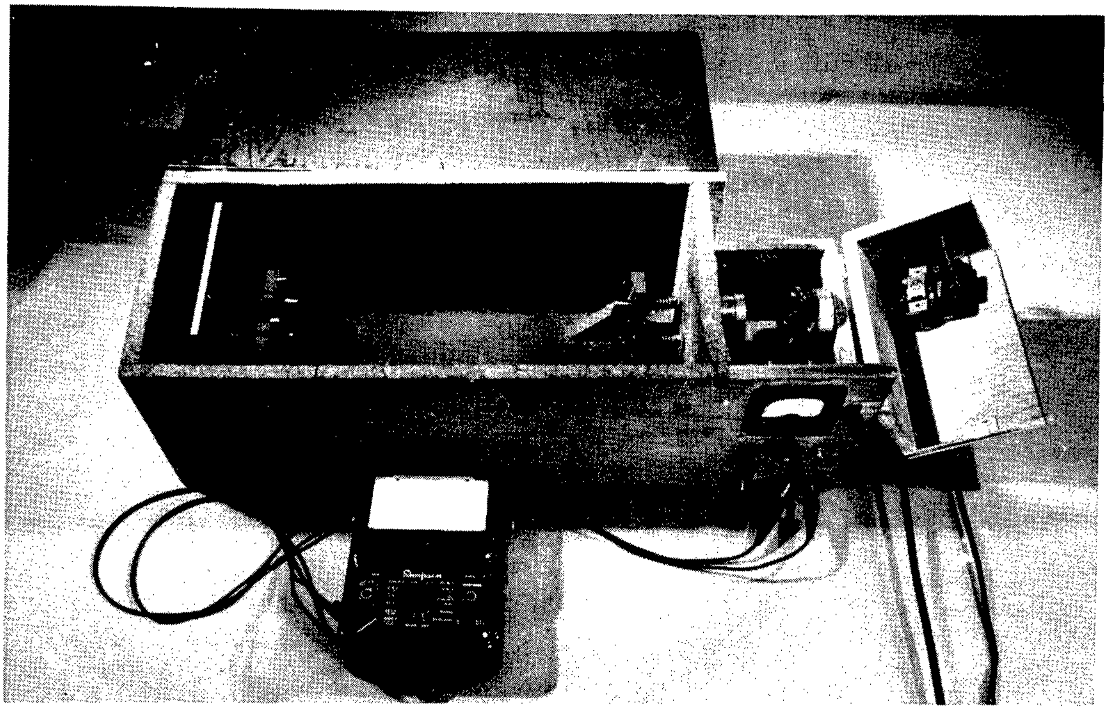

Figure 6. Haze and Light Transmittance Meter. The cover has been raised to show the internal construction.

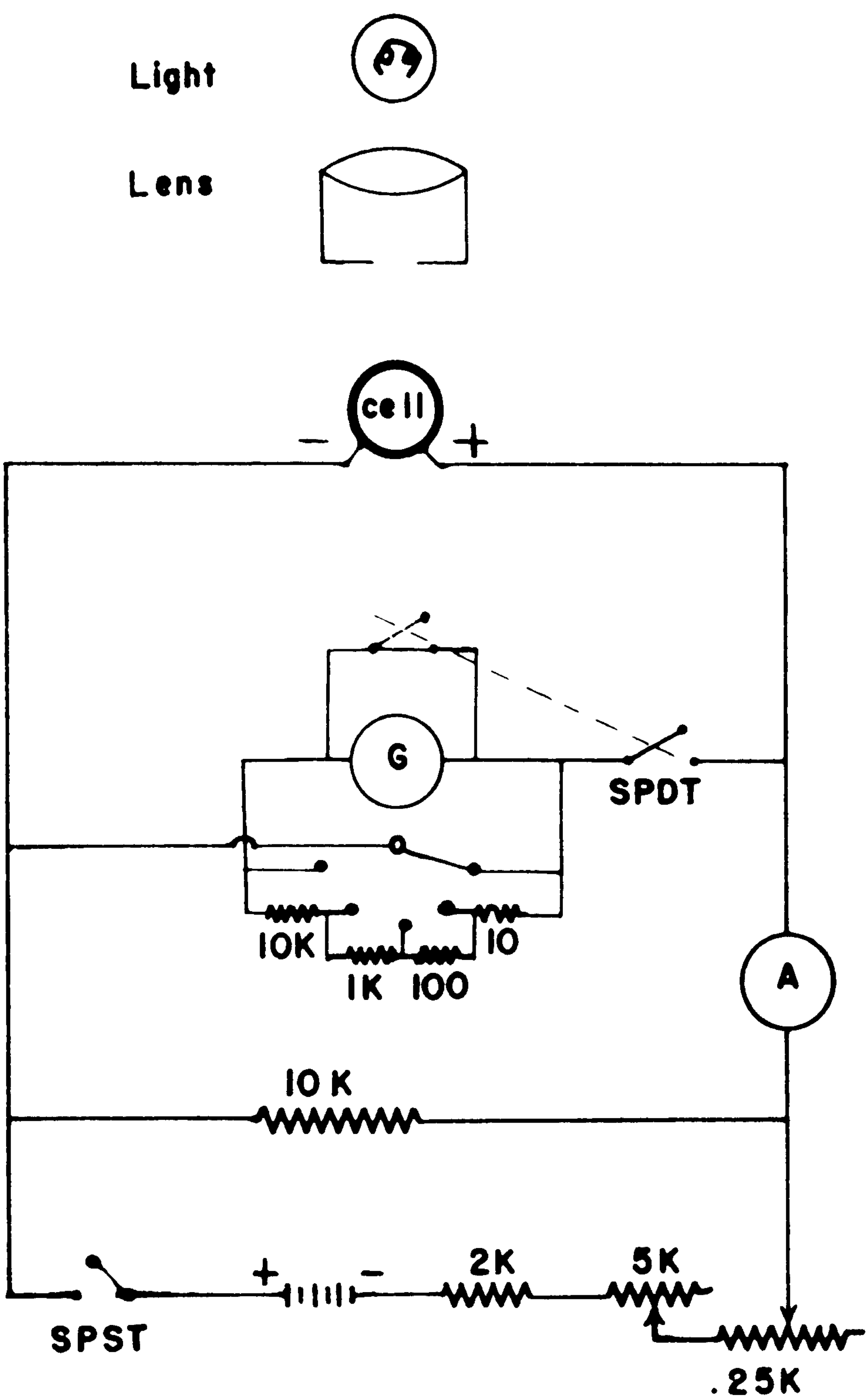

Figure 7. Schematic Drawing of the Optical and Electrical Systems of the Haze and Light Transmittance Meter.

To measure percent light transmittance, a balanced current reading is taken with the full light of the lamp incident upon the photoelectric cell. The sample is then inserted immediately before the cell aperture and a new balanced current reading taken. The percent transmittance is obtained by comparison of these two readings. Readings are taken at 3 or 4 positions on the sample to give a more reliable average value for this measurement.

To measure the haze factor, the sample is inserted against the lens aperture and a third current reading taken. The difference between this reading and that taken with the sample at the cell aperture is divided by the latter reading and the result multiplied by 100. This gives the percent of haze. The average of 3 or 4 readings is taken to give a more representative value.

2. On a Coated Canopy (Figure 8)

For measuring percent light transmittance on a canopy, two readings on a General Electric Exposure Meter are compared. The hood of the meter is replaced by a shield which is pressed against one surface of the canopy while a constant-intensity light source (incandescent lamp) is placed against the opposite surface. The lamp is connected through a constant voltage regulator and a rheostat to give the proper light intensity.

In making a test, the meter is first placed directly over the lamp at the same distance as during measurement through the canopy and the rheostat is adjusted to give full-scale reading on the meter. The meter and lamp are then placed against opposite surfaces of the canopy, as in Figure 8, and a new reading taken. The ratio of these two readings is multiplied by 100 to give percent transmittance. Measurements are made at a number of points on the canopy to show the range of values and to yield a mean value for this determination.

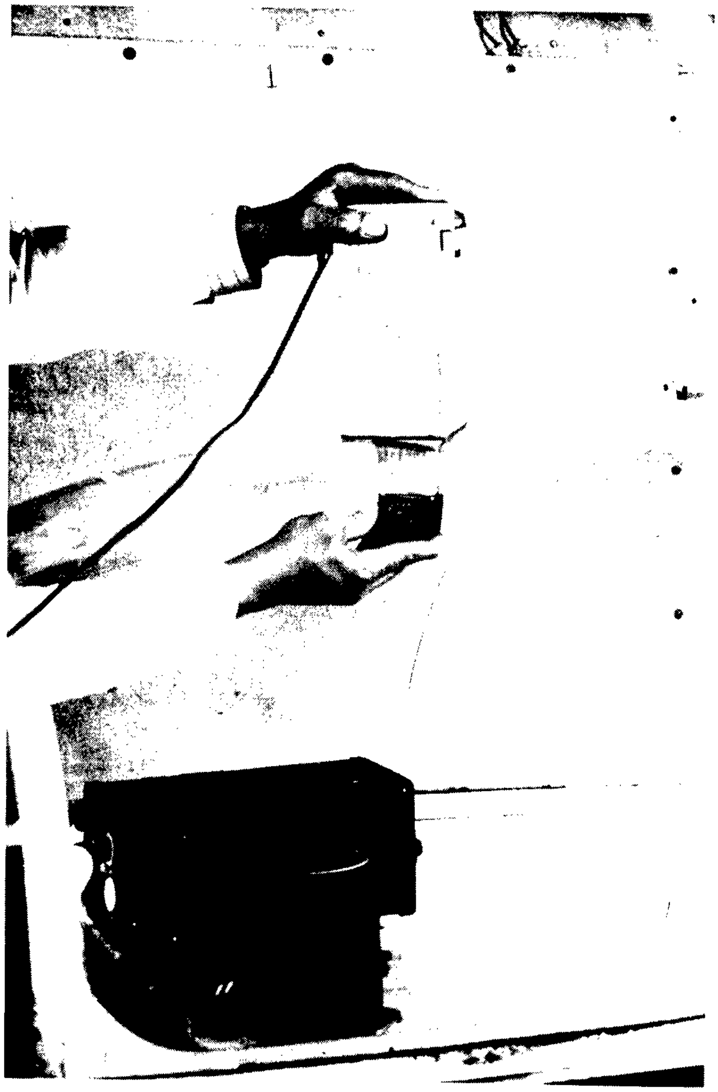

Figure 8. Measurement of Light Transmittance on Aircraft Canopy.

C. Resistance to Accelerated Weathering

The apparatus for this test, consisting of a fog chamber and sun lamp, was made for exposing specimens in accordance with Method 6021 of Federal Specification L-P-406a. (Figures 9 and 10).

The fog chamber is a 10"x 21"x 16" closed box with glass rods to support the samples. The spray nozzle delivers about 6 lb. of water spray per hour. A baffle protects the specimens from the direct impact of the spray.

The sun lamp is a Westinghouse RS-2 (275 watt). The rotating disk holding the specimens 6-1/2 inches from the center of the face of the lamp was rotated at 33 rpm.

The samples were put through ten irradiation-fog exposure cycles for a total of 240 hours. Each cycle includes 2 hours of fog, 2 hours of irradiation, 2 hours of fog, and 18 hours of irradiation. The cycling was usually continuous except for interruption to make intermediate tests on the samples.

Electrical resistance, light transmittance, haze, hardness, and other measurements were made on samples before and after exposure to determine the weather resistance of various coatings.

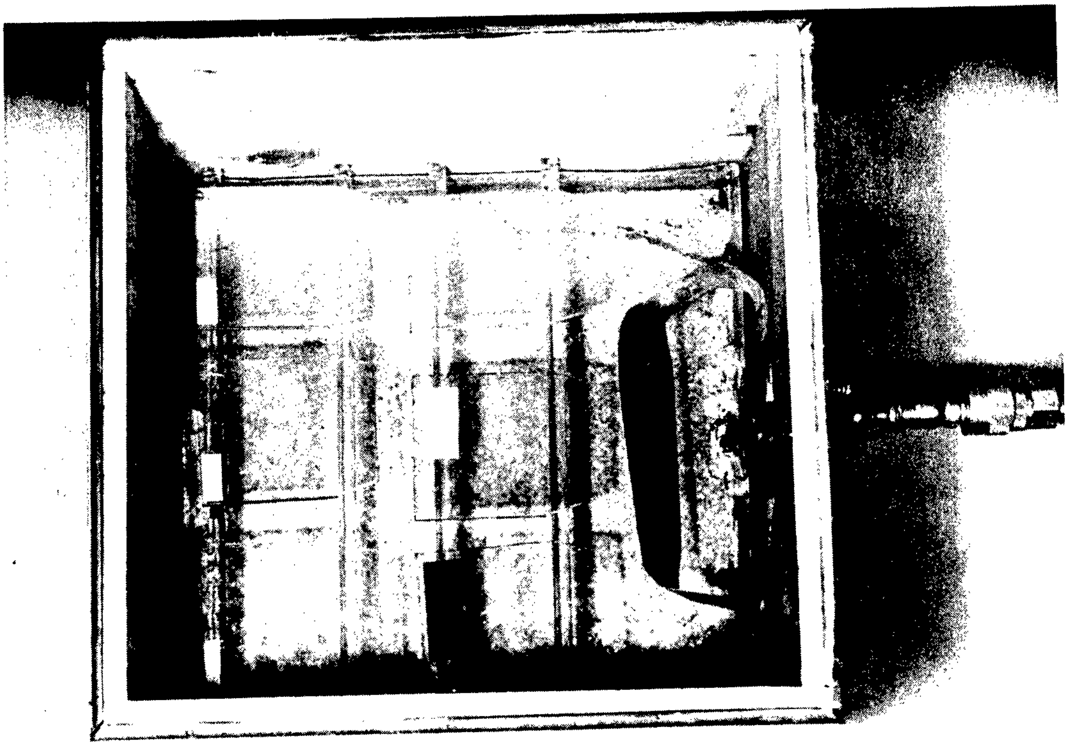

Figure 9. Top View of Fog Chamber for Accelerated Weathering. Six Specimens Are in Position.

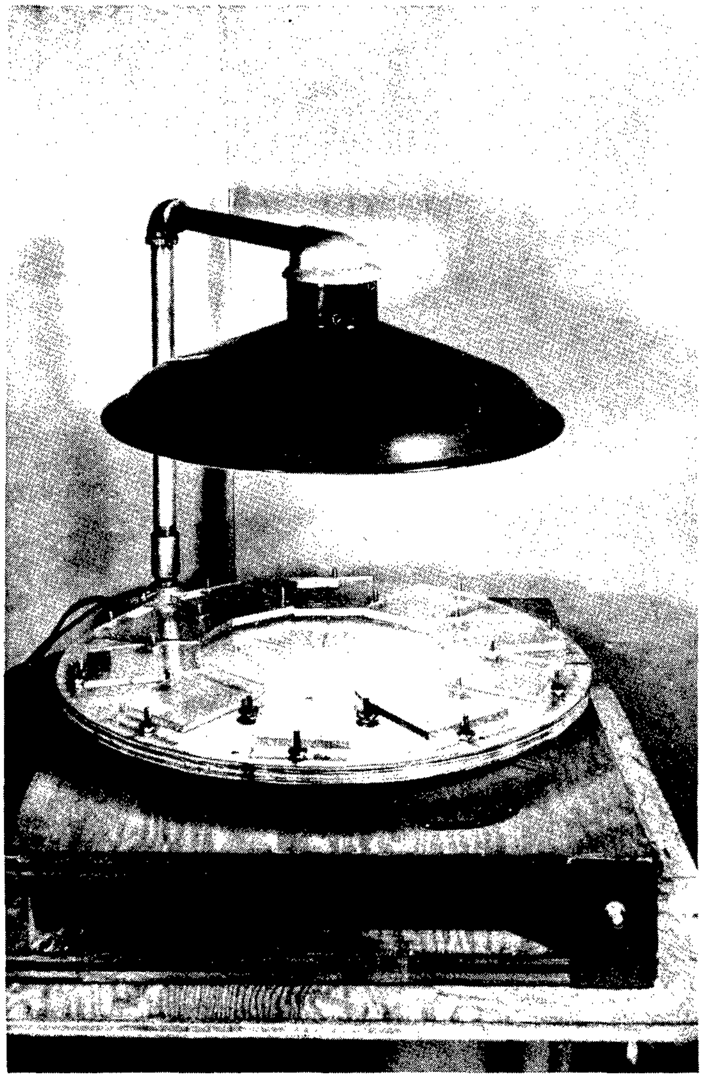

Figure 10. Irradiation Device for Accelerated Weathering. Ten Specimens Are in Position.

D. Resistance to Polishing

The coatings were tested for resistance to polishing when abraded with Federal Specification AN-C-154 Compound, Polishing (For Acrylic Plastic) using the procedure specified in paragraph F-5c of that specification. The polishing compound used was Simoniz Liquid Kleener.

E. Adhesion of the Coating

The adhesion of the coating was tested by cutting cross-marks through the film, applying pressure-sensitive tape firmly over this portion of the surface and stripping the tape away with both a smooth pull and a sudden jerk.

F. Mar Resistance and Hardness

As an additional evaluation of overcoating lacquers, the mar resistance and hardness of these films were determined.

Mar resistance was evaluated by a modification of Method 1093, Federal Specification L-P-406a. Specimens were abraded with the device in Figure 11 by the action of 400 grams of No. 80 silicon carbide in 100 gram portions. The extent of marring was measured after each portion by measuring the light transmittance and haze values, as in B above, instead of the gloss values specified in Method 1093.

Hardness was determined directly with the Sward Hardness Rocker. (Figure 12). With the rocking device resting on the test surface, the number of oscillations from a standard starting time is measured; the higher the number of oscillations the harder the surface.

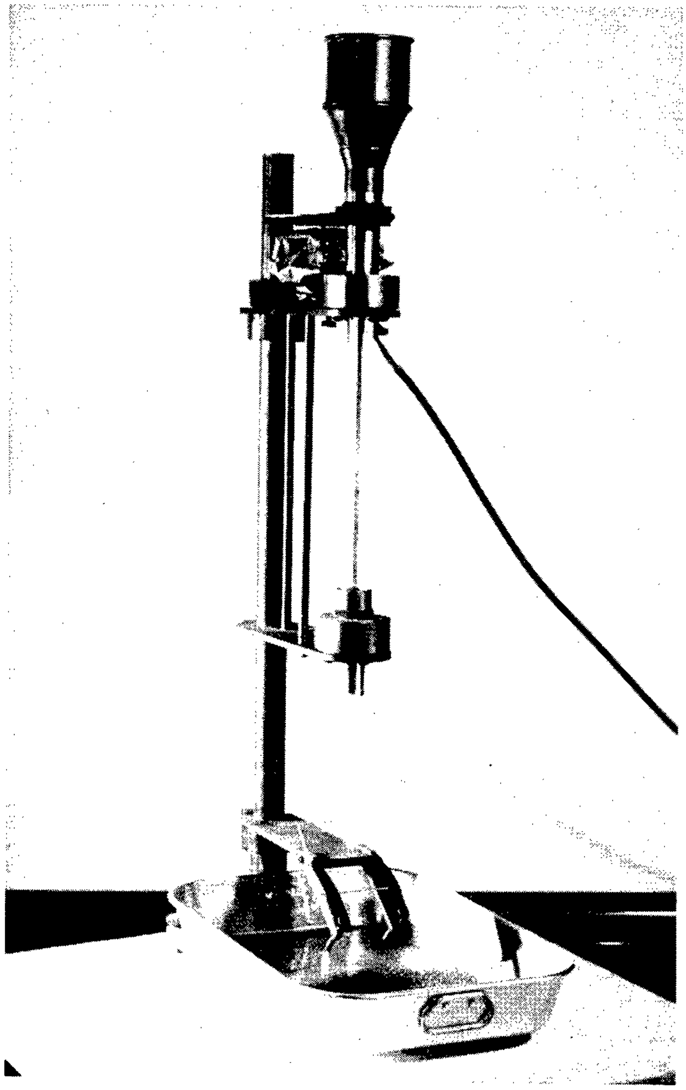

Figure 11. Abrader, for Abrading Surfaces by Falling Silicon Carbide. A specimen is in position between the clamps over the receiving pan for collecting the silicon carbide.

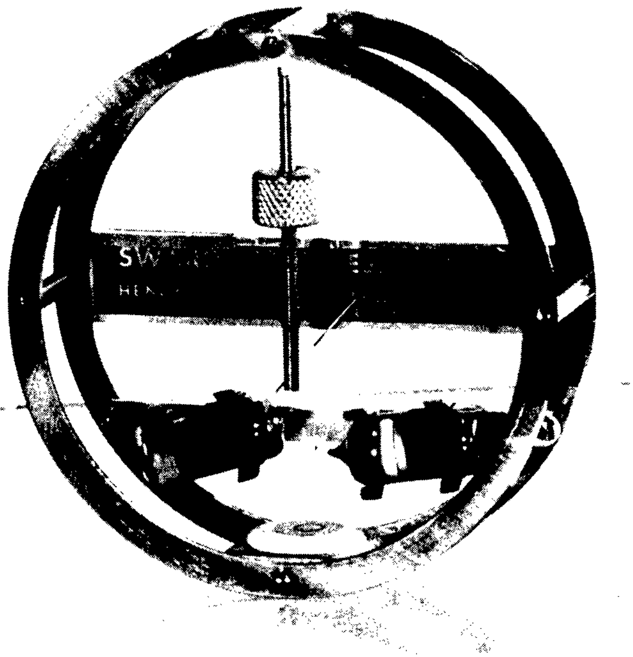

Figure 12. Sward Hardness Rocker on Plate Glass.

G. Tensile, Flexural, and Impact Strengths

The procedures for evaluating and comparing the tensile, flexural, and impact strengths of both coated and uncoated specimens were in accordance with Federal Specification L-P-406a:

Tensile strength	Method 1011
Flexural strength	Method 1031
Impact strength	Method 1071

Specimens were cut from 1/4" thick Plexiglas II sheets after portions of these sheets had been treated beforehand by a coating procedure involving random abrasion. Other portions were cut as control (untreated) specimens. The coated and uncoated specimens were given the same annealing and conditioning treatments to insure the validity of the comparisons.

H. Dielectric Loss

The original apparatus was designed for the resonant circuit, susceptance variation method using the change of capacitance procedure described in Method 4021 of Federal Specification L-P-406a. Essentially, the equipment (Figure 13) consisted of a plate oscillator tuned to 150 megacycles, a Hickok, Model 209a, vacuum tube voltmeter to measure voltage, and two pairs of 0.250 inch parallel plates, 3" diameter, made of cast aluminum and mounted on a heavy steel plate. A micrometer was mounted on one of the movable plates and the other was spring mounted to hold the specimen. Specimens were cut to three inch diameters and coated with thin aluminum foil.

Trial test runs showed evidence of lead inductance, and caused great difficulty in tuning the circuit to resonant voltage peaks with the specimen removed. A considerable voltage was read with the vernier plates shorted together, showing the inductive effect.

It was decided to switch to the change of voltage procedure with the equipment shown in Figure 14. This procedure required only one set of parallel plates.

The sizeable error caused by lead inductance was minimized by measuring the voltage directly across the condenser plates and by bringing voltage and current leads out separately. This also had the effect of sharply defining the resonance peak. As a result, the lower parallel plate capacitor was eliminated and a trimmer condenser was substituted, since the latter gave a much finer adjustment at resonance.

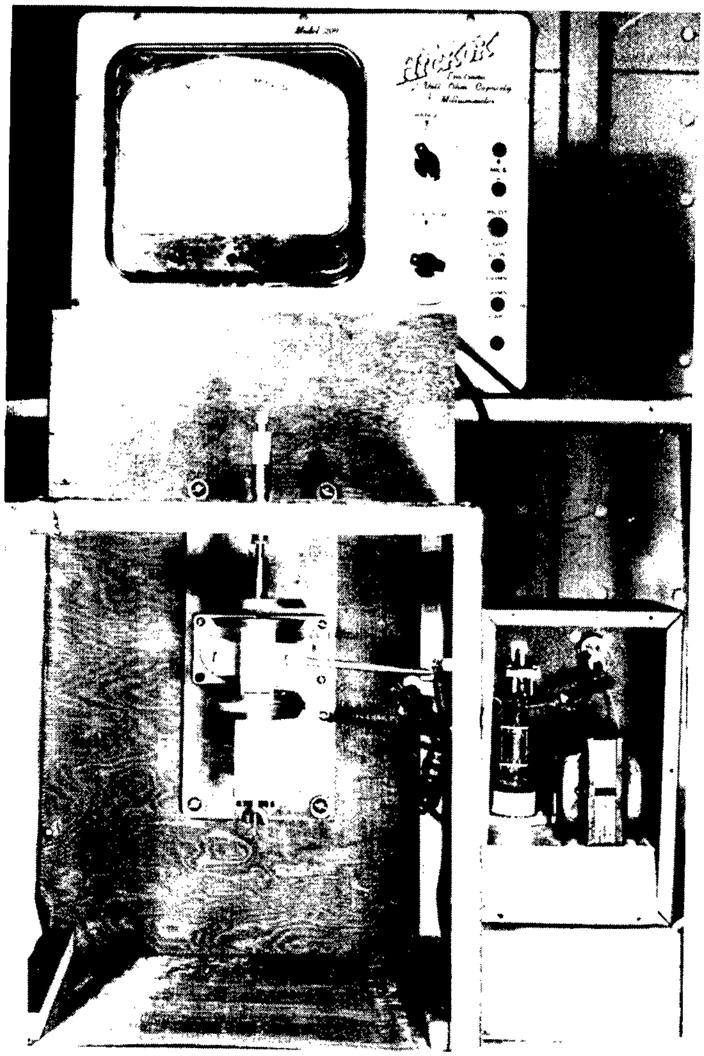

Figure 13. Original Apparatus for Measuring Dielectric Loss

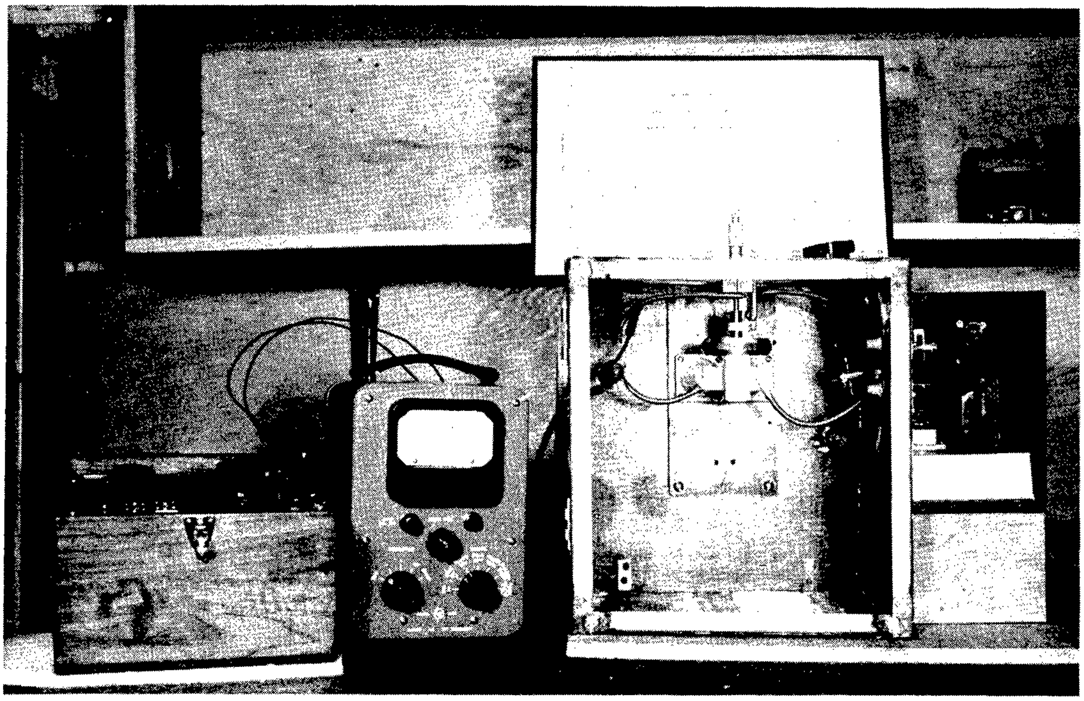

Figure 14. Improved Apparatus for Measurement of Dielectric Loss.

A Hewlett-Packard vacuum tube voltmeter, Model 410B, was used to measure the voltages. This meter is designed to read voltages accurately at frequencies up to 700 megacycles.

The following test procedure was used:

1) Clamp the specimen in the parallel plate condenser C_2.

2) Tune to resonance at desired frequency with C_t (trimmer condenser).

3) Record the resonant voltage V.

4) Remove the specimen.

5) Retune to resonance with C_2 (parallel plates), recording the resonant voltage V_o and the capacitance of the parallel plate condenser C_2 at this setting (C_r).

6) Detune on each side of resonance to 0.707 V_o and record corresponding capacitances C_o and C_o'.

$$D = \text{tangent}\,\delta = \frac{\Delta C_o}{2\,C_p} \times \frac{V_o - V_1}{V_1}$$

where D is the dissipation factor
δ is the loss angle

$$\Delta C_o = C_o - C_o'$$

$$C_p = C_r - C_2' + C_a'$$

where $C_r - C_2'$ can be found from the calibration curve, Figure 15.

C_p = capacitance of specimen.

C_r = calibration capacitance of parallel-plate condenser C_2 at second resonant adjustment.

C_2' = calibration capacitance of the parallel-plate capacitor C_2 at a setting equal to measured thickness of specimen dielectric.

C_a' = calculated capacitance of C_2 at setting equal to thickness of specimen.

Repeated tests with the same specimen showed variations in the measured dielectric loss. This variation was due to reading the small difference between the voltage at resonance with and without the specimen ($V_o - V_1$). The small voltage difference (about 0.2 volt) was read much more accurately by use of a potentiometer connected directly across the meter movement. There is no loading on the meter when the potentiometer is balanced, and use of the potentiometer gives a much more sensitive meter movement combined with a greatly expanded scale. The result is that measurements can be made accurately to within 0.01 volt by use of the potentiometer, as compared to 0.1 volt without it. With the voltage difference being of the order of 0.2 volt, this measurement can now be made to within about 5% accuracy.

The parallel plates used in the change of voltage procedure were calibrated with a capacitance bridge at audio frequencies. The results were then plotted in a curve of capacitance versus distance between plates read on the micrometer. (Figure 15). In this procedure, the absolute value of capacitance is immaterial, since only the capacitance difference between the two micrometer settings ($C_r - C_2$) is of importance. It is for this reason that the curve approaches a fixed value of 45 mmf as the condenser plates are moved far apart. When used for a change in capacitance resulting from a change in plate separation, the curve gives very accurate capacitance differences.

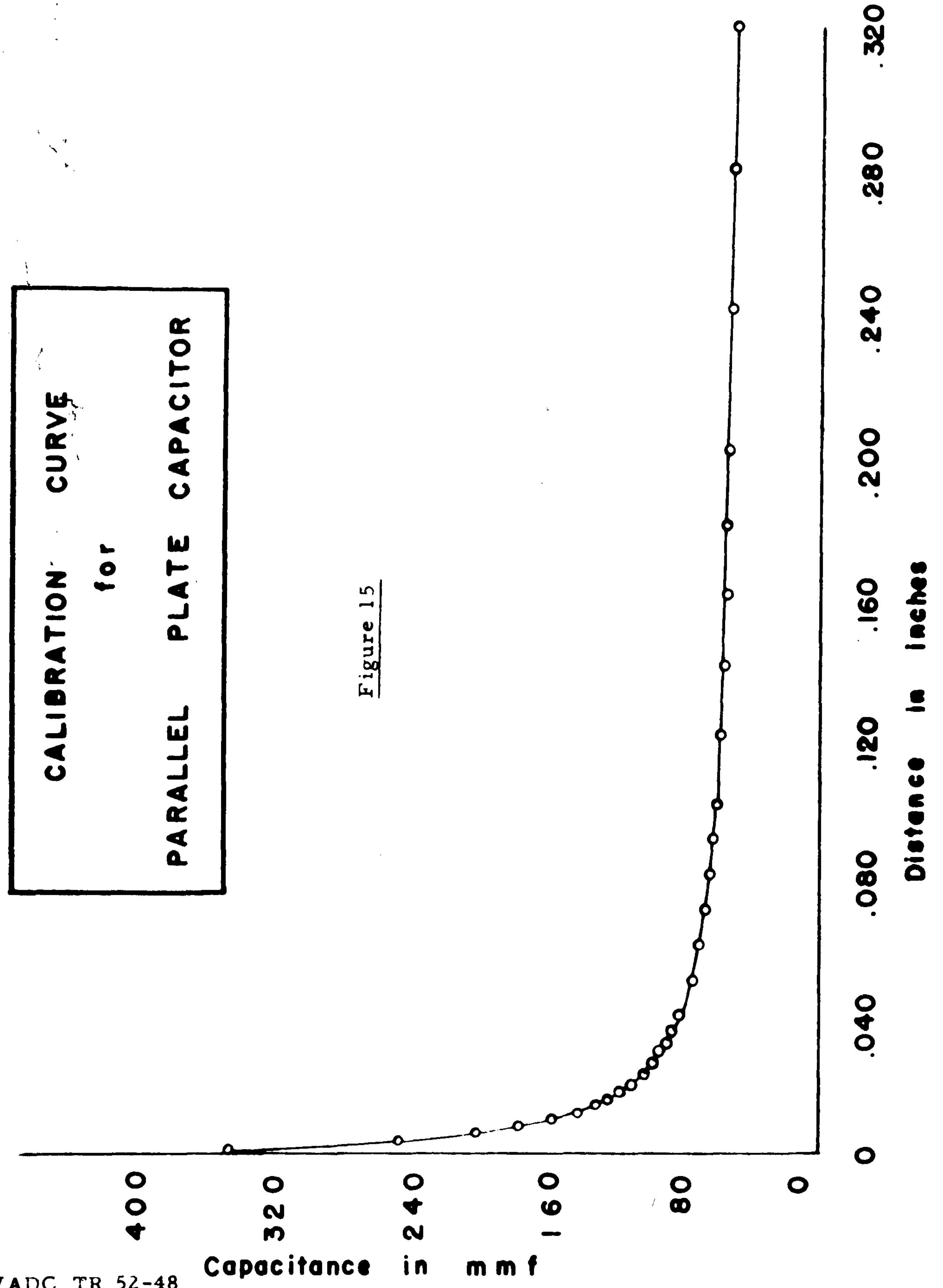
CALIBRATION CURVE
for
PARALLEL PLATE CAPACITOR
Distance in Inches
0
.040
.080
.120
.160
.200
.240
.280
.320
Capacitance in mmf
0
80
160
240
320
400

Figure 15

SECTION IV

TEST RESULTS

Specimens coated with graphite and sprayed with the protective overcoating were tested in accordance with the procedures in Section III.

A. Surface Resistance and Accelerated Weathering

Test panels, 3" x 3", were exposed to 240 hours of accelerated weathering and their surface resistances measured at various times. (Table No. 3). These results indicated that the applied coating (graphite and ES-4) was resistant to the effects of weathering and that, under controlled conditions of application, a surface resistance of 10 megohms or less could be maintained. Table No. 4 compares various overcoatings with the recommended ES-4.

There was no evidence of cracking, peeling, crazing, or any other surface instability.

B. Haze and Light Transmittance

As shown in Table No. 5, weathering for 240 hours has no significant effect on the haze and light transmittance of specimens coated with the overcoatings used in this work.

C. Resistance to Polishing

After coated specimens had been polished with Simoniz Liquid Kleener, there was no evidence of removal of the overcoating or the graphite. The values for surface resistance, haze, and light transmittance of these specimens, after polishing, did not vary significantly from those measured before polishing.

TABLE NO. 3

SURFACE RESISTANCES OF COATED METHACRYLATE SPECIMENS

EXPOSED TO ACCELERATED WEATHERING TEST

(In Megohms Per Square)

	Sample Number						
	0-601	0-602	0-603	0-604	0-605	0-606	0-607
Before Over-Coating	4	1-2.5	1-2	2.5	5	0.8-1.2	1.5-2.0
After Over-Coating	5	1.5-3	2-4	3-5	7-10	1.0-1.8	1.0-1.4
After Weathering 3 hrs.	5	1.5-3	2-4	3-5	7-15	1.0-1.8	1.2-1.7
6 hrs.	7	1-3	2-4	3-5	7-15	0.8-1.6	1.2-1.8
12 hrs.	7-10	1-4	2-4	3-5	7-15	1.0-1.6	1.2-2.0
24 hrs.	6-10	1-4	3-4	3-5	8-15	1.6	1.3-2.0
48 hrs.	6-8	1-3.5	3-4.5	3.5-5	8-16	1.4-1.8	1.2-2.2
96 hrs.	6-10	1.5-4	3-5	4-6	11-17	1.0-1.6	1.2-2.4
192 hrs.	6-11	1.8-5	3-5	4-6	11-19	1.2-1.8	1.4-2.6
240 hrs.	8-11	1.6-4.5	4-6	5-6	10-18	1.0-1.8	1.4-3.0

TABLE NO. 3 (Cont.)

SURFACE RESISTANCE OF COATED METHACRYLATE SPECIMENS EXPOSED TO ACCELERATED WEATHERING TEST

(In Megohms Per Square)

	Sample Number						
	0-608	0-609	0-610	0-611	0-612	0-613	0-614
Before Over-Coating	0.6-1.0	1.0-1.6	0.8-1.2	2	3	2-3	2-3
After Over-Coating	0.4-0.8	0.7-1.4	0.6-1.0	2-3	3-5	5-10	3-4
After Weathering 3 hrs.	0.6-0.8	0.8-2.6	0.8-1.0	2-3	3-5	5-10	3-5
6 hrs.	0.5-0.8	0.8-2.0	0.5-1.0	2-3.5	5-8	6-10	3.5-5
12 hrs.	0.6-1.0	0.8-2.5	0.6-1.0	2.0-3.5	6-10	6-10	4-6
24 hrs.	0.7-1.1	1.2-3.0	0.8-1.2	2-3.5	7-10	6.5-10	4-6
48 hrs.	0.6-1.0	1.2-3.2	0.8-1.2	2.5-4	8-12	7-10	5-6
96 hrs.	0.6-1.0	1.0-5.0	0.8-1.2	2-4.5	6-15	9-14	6-9
192 hrs.	0.8-1.2	1.2-5.0	1.0-1.6	3-5	13-19	9-19	6-9
240 hrs.	0.8-1.4	1.2-5.0	0.8-1.2	3-5	15-20	9-20	6-10

TABLE NO. 3 (Cont.)

SURFACE RESISTANCE OF COATED METHACRYLATE SPECIMENS EXPOSED TO ACCELERATED WEATHERING TEST

(In Megohms Per Square)

	Sample Number					
	0-615	0-621	0-622	0-623	0-624	0-625
Before Over-Coating	1-2	2-3	2-3	2-3	1-2	0.2-0.8
After Over-Coating	1-2	4-9	4-10	3-5	1.5-5	0.5
After Weathering 3 hrs.	2-3	4-9	7-10	3-5	2-4	0.5
6 hrs.	2-3	5-12	6-18	3-6	2-7	0.4
12 hrs.	2-3.5	6-15	9-20	3-7	2-7	0.4-0.6
24 hrs.	2-4	6-15	9-20	3-8	2-7	0.2-0.6
48 hrs.	2-4	8-18	9-21	4-9	2.5-7	0.3-0.7
96 hrs.	3-5	10-22	10-40	3-9	3.5-9	0.4-0.8
192 hrs.	4-6	12-29	16-55	6-12	5-15	0.5-0.9
240 hrs.	3-6	12-28	19-60	4.5-12	4-11	0.6-0.8

Nos. 0-601 - 0-605 exposed only to sunlamp for 200 hours.
Nos. 0-611 - 0-615 exposed to fog only for 40 hours.
Others exposed to 200 hours sunlamp plus 40 hours in fog box.

TABLE NO. 4

SURFACE RESISTANCE OF COATED METHACRYLATE SPECIMENS WITH VARIOUS OVERCOATINGS

(Exposed To Accelerated Weathering Test - In Megohms Per Square)

Sample No.	Over-Coating	Before Over-Coating	After Over-Coating	After Weathering			
				24 hrs.	96 hrs.	192 hrs.	240 hrs.
0-666	ES-6	0.3-0.6	2.4-5.0	3.0-5.5	2.6-5.5	3.6-8.0	4.2-9.0
0-667		0.3-0.4	0.6-1.0	1.0-1.5	1.4-1.6	0.8-1.2	1.2-1.8
0-668		0.4-0.5	1.2-1.6	1.2-2.8	2.4-3.0	1.6-5.0	1.8-5.0
0-669		0.6-1.0	1.6-2.2	2.2-3.0	3.2-5.5	2.8-4.8	3.0-4.8
0-670		0.8-1.0	2.2-2.8	2.4-3.3	3.8-4.2	2.8-3.6	3.8-4.6
0-671	R-108	0.3-0.6	1-3	1.6-3.0	2.2-2.6	0.6-1.6	1.2-2.4
0-672		0.2-0.6	1-4	1.2-5.5	1.4-4.5	0.6-1.6	1.0-2.4
0-673		1.2-1.6	3.4-5	5.5-8.5	6-8	3.8-8.0	4.8-1.0
0-674		0.6-1.0	2.2-5.0	3.6-7.0	3-10	2.2-6	2.8-9
0-675		0.6-0.8	2.0-3.2	3.1-4.4	3.6-4	2.0-2.4	2.6-3.8
0-681	ES-4	0.3-0.6	0.6-1.0	0.9-1.2	1.1-1.3	1.6-1.8	1.6-1.8
0-682		0.6-0.8	0.6-1.0	0.5-0.6	0.6-0.8	0.6-0.8	0.8-1.2
0-683		1.1-1.3	1.0-1.4	0.9-1.1	1.0-1.4	1.2-1.8	1.2-1.6
0-684		0.8-1.2	1.0-1.4	1.1-1.4	1.1-1.8	1.2-1.8	1.4-1.8
0-685		0.5-0.8	0.8-0.9	0.8-1.1	1.0-1.2	1.0-1.6	1.2-1.6
0-686	ES-11	0.5-1.1	0.8-1.4	0.8-1.3	0.8-1.4	1.0-1.6	1.1-2.0
0-687		0.3-0.6	0.4-0.6	0.6-0.8	0.6-1.0	0.6-1.0	0.6-1.2
0-688		0.2-0.6	0.7-1.0	0.6-1.1	0.8-1.0	1.0-1.4	0.9-1.2
0-689		0.9-1.6	1.2-2.4	1.0-1.9	1.0-1.7	1.4-1.8	1.2-2.0
0-690		0.7-1.7	2.6-3.4	1.4-3.2	2.5-3.5	2.4-4.0	3.0-4.6
0-731	ES-14	0.4-0.8	0.6-1.0	0.7-1.2	0.8-2.0	0.3-12.0	3.6-12.0
0-732		0.7-1.0	0.9-1.1	1.2-1.4	2.0-2.2	5-10	7-15
0-733		0.4-0.8	0.6	0.5-0.8	0.6-0.8	1.2-1.4	1.4-1.6
0-734		0.4-1.0	0.6-0.9	0.9-1.2	0.7-1.2	1.6-2.6	1.6-3.6
0-735		0.5-0.8	0.8-1.0	0.8-1.2	0.8-1.2	1.8-5.0	2.4-3.2
0-736	ES-4	0.3-0.4	0.4-0.6	0.8-1.0	0.4-0.6	0.6-0.7	0.5-0.6
0-737		0.2-0.7	0.2-0.6	0.2-0.7	0.2-0.7	0.2-0.7	0.2-0.6
0-738		0.3-0.5	0.5-0.6	0.9-1.1	0.7-0.8	0.6-0.8	0.6-1.1
0-739		0.3-0.5	0.3-0.6	0.4-1.0	0.3-1.3	0.4-1.2	0.3-1.5
0-740		0.8-1.2	0.8-1.2	1.1-1.3	1.0-1.2	1.1-1.3	1.0-1.3

TABLE NO. 5

LIGHT TRANSMITTANCE AND HAZE VALUES OF COATED SPECIMENS

(Weathered for 240 Hours)

Sample No.	Overcoating	Before Weathering		After Weathering	
		% Light Transmittance	% Haze	% Light Transmittance	% Haze
0-666	ES-6	87.8	2.6	86.4	2.1
0-667		85.5	3.5	84.0	2.5
0-668		86.4	2.7	85.2	2.1
0-669		87.7	1.1	86.1	2.1
0-670		86.8	2.3	86.4	2.8
0-671	R-108	84.4	1.6	84.0	2.5
0-672		84.7	1.6	82.2	2.6
0-673		87.0	2.3	86.1	2.8
0-674		85.7	2.3	82.8	4.4
0-675		84.3	0.8	82.8	4.0
0-681	ES-4	85.7	3.1	87.0	4.1
0.682		90.3	2.6	90.6	2.0
0-683		88.7	2.6	89.3	1.0
0-684		89.0	1.9	89.9	1.3
0-685		87.7	2.3	88.9	2.7
0-686	ES-11	87.7	2.3	88.0	2.4
0-687		89.4	1.5	88.9	1.7
0-688		88.7	1.5	89.8	3.4
0-689		86.7	3.5	87.0	6.2
0-690		89.3	2.2	90.6	3.3
0-731	ES-14	79.8	4.6	78.9	7.7
0-732		81.5	3.4	79.2	7.6
0-733		80.6	1.8	79.1	8.8
0-734		83.5	2.7	81.8	7.0
0-735		86.4	1.3	82.1	2.2
0-736	ES-4	78.7	4.3	78.5	5.0
0-737		75.9	3.0	76.1	5.2
0-738		78.9	1.8	78.7	2.3
0-739		77.8	3.7	77.8	4.3
0-740		81.7	3.2	82.4	3.7

D. Adhesion of the Coating

When pressure-sensitive tape pressed to the coated surface was stripped away, the coating was not removed from the surface. Measurements of the surface resistance, haze, and light transmittance did not vary significantly before and after such treatment.

E. Mar Resistance

The extent of marring was measured with the equipment described previously by evaluating the surface resistance, haze, and light transmittance of coated specimens before and after abrasion with silicon carbide. (Table No. 6). The surface resistance of the specimens did not increase significantly and the decrease in light transmittance was comparable. There was considerable variation in the increase in haze with the ES-4 overcoating showing the lowest average increase (540%). The average haze increase for Plexiglas II sheet acrylic (1481%) was the highest for any of the materials, indicating less mar resistance than any of the coated specimens.

F. Hardness

As compared with plate glass as the standard, there is little difference in hardness between the coated specimens and uncoated Plexiglas II. Direct comparison with Plexiglas II indicates that the coated specimens have a surface slightly less hard. (Table No. 7).

G. Tensile, Flexural and Impact Strengths

There were no significant differences in the tensile, flexural, and impact strengths of uncoated and coated specimens. (Table No. 8).

TABLE NO. 6

SURFACE RESISTANCE, LIGHT TRANSMITTANCE, AND HAZE VALUES FOR COATED METHACRYLATE SPECIMENS

(Before and After Abrasion With 400 Grams Silicon Carbide)

Sample No.	Over-Coating	Resistance, Megohms/Square		% Light Transmittance		% Haze		
		Before	After	Before	After	Before	After	% Increase
0-696	ES-4	0.9	1.4	90.1	82.7	2.9	19.8	583
0-697		0.6	1.0	90.4	87.5	2.6	24.5	842
0-698		0.4	0.7	85.1	79.8	4.0	22.8	470
0-699		0.5	0.7	83.6	77.1	3.0	11.0	267
0-702	R-108	1.8	3.3	84.3	75.5	5.2	27.7	433
0-703		7.5	8.7	90.3	86.2	1.7	30.9	1718
0-704		3.7	4.8	90.8	84.7	3.2	20.1	528
0-705		2.0	2.2	85.4	80.7	2.0	12.3	515
0-707	ES-6	0.7	1.3	89.3	84.6	2.2	14.9	577
0-708		2.2	5.2	91.8	83.6	1.4	21.4	1429
0-709		1.6	3.4	91.8	87.1	1.8	20.9	1061
0-710		2.2	4.2	89.9	86.0	1.9	17.9	842
0-712	ES-11	0.6	2.1	89.8	84.8	4.1	25.0	510
0-713		0.7	3.0	89.4	85.5	2.2	31.2	1318
0-714		1.1	2.5	90.1	86.8	2.7	35.8	1226
0-715		2.4	2.5	90.7	85.9	1.9	34.2	1700
0-717	ES-14	0.8	1.1	89.2	85.6	2.2	17.5	695
0-71		2.1	3.1	90.2	85.5	1.5	18.4	1127
0-719		0.7	1.0	88.6	86.8	3.7	23.1	524
0-720		0.7	1.5	90.1	85.6	2.6	14.1	442
0-721	ES-15	0.8	0.8	87.9	78.5	6.7	22.0	228
0-722		1.4	1.6	83.3	80.3	1.6	23.0	1338
0-723		0.8	1.7	89.8	84.4	1.9	17.1	800
0-724		1.3	1.9	88.8	85.0	1.9	18.6	868
0-727	None			94.8	90.3	0.6	7.6	1167
0-728	Plex.II			95.1	91.5	0.6	10.2	1600
0-729	sheet			95.8	92.6	0.7	13.0	1757
0-730				95.8	90.5	0.7	10.5	1400

TABLE NO. 7

SWARD (ROCKER) HARDNESS VALUES OF COATED PLEXIGLAS, UNCOATED PLEXIGLAS, AND PLATE GLASS

Sample Number	Overcoating	Hardness Value (Average of 10 values)
0-696	ES-4	36.6
0-697		35.8
0-698		34.4
0-699		32.4
0-700		33.2
0-741		40.4
0-742		38.8
0-743		40.4
0-744		36.8
0-745		29.8
0-701	R-108	37.0
0-702		34.6
0-703		40.8
0-704		38.0
0-705		33.0
0-706	ES-6	39.0
0-707		39.0
0-708		43.2
0-709		38.0
0-710		35.8
0-711	ES-11	44.6
0-712		40.4
0-713		46.2
0-714		39.8
0-715		44.6
0-721	ES-15	37.6
0-722		37.0
0-723		42.0
0-724		38.2
0-725		38.6

TABLE NO. 7 (Cont.)

SWARD (ROCKER) HARDNESS VALUES OF COATED PLEXIGLAS, UNCOATED PLEXIGLAS, AND PLATE GLASS

Sample Number	Overcoating	Hardness Value (Average of 10 values)
0-716	ES-14	40.6
0-717		39.0
0-718		41.2
0-719		36.2
0-720		32.8
0-746		39.2
0-747		41.8
0-748		41.0
0-749		38.2
0-750		33.8
0-726	None	41.6
0-727	Plexiglas II	38.4
0-728	Control	42.8
0-729		40.4
0-730		44.2
0-751		38.0
0-752		42.8
0-753		44.6
0-754		43.8
0-755		41.2
I	None	101.2
I	Glass	100.0
I		108.8
I		111.0
I		105.2
I		104.0

TABLE NO. 8

COMPARISON OF TENSILE, FLEXURAL, AND IMPACT STRENGTHS OF UNCOATED AND COATED ACRYLIC

Type of Specimen	Tensile (psi)	Flexural (psi)	Impact (Izod) (ft. lb. /in.)
Uncoated	10,800	1744	52
Coated	10,700	1720	53

Note: All values are averages for 3 to 10 samples.

H. Dielectric Loss Factor

Final tests were made on three circular coated specimens, 1/4" thick, with a resistance of 1-10 megohms per square. The specimens were coated with a thin layer of aluminum foil. The average value of D, the dissipation factor, for a series of tests was between 0.007 and 0.011.

Since

$$\text{Loss factor} = KD$$

where K = dielectric constant (1.6 for Plexiglas)

$$D = \tan \delta$$

the average loss factor was between 0.0112 and 0.0172. These values are slightly above the specified 0.01 loss factor.

With uncoated Plexiglas II, the average loss factor was 0.0112 so that the increase in the dielectric loss factor was considerably below 0.01 at 150 mc. For more extensive use of the equipment and more precise measurements in evaluating the dielectric loss factor, standardization of the procedure and further elimination of electrical pickup would be necessary.

SECTION V

COATING OF AIRCRAFT CANOPIES

Three canopies, one used and two new ones, were supplied by the Air Force and were coated under laboratory conditions. One of the new canopies has performed satisfactorily in flight tests at Wright Air Development Center.

A. Treatment of New Canopies

The canopy was abraded by rubbing with a brush or abrasive pad, using a rotary motion which made random scratches over the entire surface. The lower edge of the canopy, which was too hard to be scratched sufficiently by the pad, was abraded with very fine sandpaper.

Graphite was then applied by the use of a soft graphite-saturated cloth which was rubbed uniformly and firmly in all directions over the surface to cover every spot with a number of strokes in different directions.

The surface was next rubbed with a clean cloth pad, using more pressure than in the previous operation. This gave a smoother, more uniform coating with increased adhesion of the graphite. It also removed excess graphite from some areas - greatly improving optical clarity at these spots. The entire surface was brushed off to remove loose graphite.

The canopy was then sprayed with a thin even overcoating of the methacrylic acid-methyl methacrylate copolymer formulation ES-4.

Measurements of surface resistance and light transmittance were made at a large number of points on the coated canopy. The average resistance was 2 megohms/square before application of the overcoating and 6 megohms/square after application, with a range of 2-10 megohms. The light transmittance values ranged from 85 to 91% before overcoating and from 79 to 87% afterwards.

B. Treatment of Used Canopy

A used canopy assembly taken from service at the Mobile Air Material Area was coated similarly with a variation in the abrasion operation.

Because of the extensive scratching and marring already present on the canopy, it was first washed with water containing a detergent and then cleaned with Du Pont No. 7 Polish and Cleaner. Without the usual abrasion step, it was coated with graphite as above. A fairly uniform coating with a few dark smudges was obtained. The electrical resistance was between 0.5 and 10 megohms/square but the light transmittance was too low, ranging from 70 to 85%.

This coating was, therefore, removed by polishing the canopy with Simoniz Liquid Kleener and graphite applied again. Again the resistance ranged from 0.5 to 10 megohms/square but the light transmittance was now satisfactory, ranging from 80 to 90%.

Within a few seconds after the canopy had been sprayed with the ES-4 formulation intense crazing developed on the surface of the canopy.

Experiments with small methacrylate specimens demonstrated that this crazing was due to the action of solvents contained in the spray solution on the canopy surface which was under tension from being fitted and assembled into the canopy frame. When these small specimens were bent to about the same curvature as the formed canopy, a large number of solvents, excluding only hydrocarbons and carbon tetrachloride, caused severe crazing. These solvents did not craze unformed specimens.

Annealing the specimens in an oven in the bent condition, at temperatures from 70 to 100°C., followed by slow cooling, 15°C. per hour, to 50°C., before application of the solvent or solution, prevented their crazing. Annealing the specimens in hot water for more effective control of the temperatures was unsuccessful. Water had a decided deleterious effect on methacrylate so that even flat specimens heated in water crazed on subsequent solvent treatment.

SECTION VI

SUMMARY AND CONCLUSIONS

A. Coating Materials and Application

The application of finely divided graphite (Dixon Airspun Graphite, Type 200-10) and a methacrylic acid - methyl methacrylate copolymer resin to acrylic plastic produced an electrically conducting transparent coating. The procedure of application included:

1. Abrading the acrylic surface in perpendicular directions with a suede brush.
2. Rubbing in the graphite with a graphite impregnated soft cloth and pressing in the graphite particles with a pad of clean soft cloth.
3. Spraying the graphited surface with a 1:5 methacrylic acid - methyl methacrylate copolymer as in overcoating.

The copolymer, identified as ES-1, was prepared by refluxing methacrylic acid and methyl methacrylate, in the molar ratio of 1:5, in a cyclohexanone solution.

The following formulation was used for spraying the graphited acrylic surface:

Copolymer ES-1	1 part
Butyl Acetate	2 parts
Methyl Ethyl Ketone	1 part

Other materials tried as overcoatings which proved unsatisfactory included:

Silver deposited in very thin layers
Silver, zinc, aluminum, and iron powders
Molybdenum disulfide, and
Titanium compounds.

As the overcoating, the following materials were investigated and proved less satisfactory than the methacrylic acid-methyl methacrylate copolymer:

Plasticized Nylon,
Acrylic-amine copolymers,
Urethans,
Metallic salts, and
Chloride-releasing polymers.

B. Properties of Coated Acrylic

The composite material, consisting of acrylic and developed coating, had the following properties:

1. Mechanical

a. The coated material had essentially the same tensile, flexural, and impact strengths, mar resistance, and hardness as the base material specified in Military Specification MIL-P-5425 for Plastic, Acrylic Sheet, Heat Resistant.

b. The adhesion of the coating was such that it was not removed when pressure-sensitive tape pressed to the surface of the composite material was stripped away.

c. The coating was not removed or visibly scratched and had a light transmittance in excess of 80% when abraded with Specification AN-C-154 Compound, Polishing (For Acrylic Plastic) using the procedure specified in paragraph F-5c of that specification.

2. Optical

a. The light transmittance of the coated acrylic was in excess of 80% when tested in accordance with Method 3021 of Federal Specification L-P-406a, Plastics, Organic, Test Methods, General Specification.

b. The haze value of all but two samples of the coated plastic did not exceed 4% when measured in accordance with Method 3021 of Federal Specification L-P-406a.

c. Surface reflections were minimized as indicated by the slight change in light transmittance before and after application of the coating. Consequently, the refractive index of the coating matched the base material very closely.

d. The thickness of the applied coating was less than 0.001 inch.

e. After exposure to the accelerated weathering test, Method 6021 of Federal Specification L-P-406a, the coated material showed no excessive cracking, peeling, crazing, or other surface instability in comparison with uncoated acrylic material conforming to Specification MIL-P-5425. The light transmittance of the coated material was in excess of 75 percent and the haze value did not exceed 5 percent after accelerated weathering.

f. The coating was non-porous so that finger marks and oil smears could be removed easily with soap and water.

3. Electrical

a. The direct current surface resistance of the coated plastic was in the range 1-10 megohms per square when measured in accordance with Method 4041 of Federal Specification L-P-406a.

b. The surface resistance of the coated plastic was relatively insensitive to wide variations in relative humidity, temperature, and atmospheric pressure conditions.

c. The average dielectric loss factor of the coated acrylic was 0.0112-0.0172 when measured as specified in Method 4021 of Federal Specification L-P-406a at frequencies up to 150 mc. The dielectric loss factor determined by this procedure for Plexiglas II was 0.0112 so that the increase resulting from the coating was less than 0.01.

Two new F-86 aircraft canopies were treated with the developed coating. One of these was tested in flight by Wright Air Development Center and performed satisfactorily. A third F-86 canopy taken out of service developed severe surface crazing on application of the overcoating solution. This was attributed to surface strains introduced into the methacrylate during assembly into the canopy frame and/or by service conditions. Similar crazing was produced experimentally in small methacrylate specimens bent to about the same curvature as the canopy. Such crazing was prevented by annealing the specimens by retaining them at 70°C. for one hour and then cooling at about 15°C. per hour to 50°C.

C. Recommended Coating Procedure

1. Application of Graphite

a. Clean the surface to be coated so that it is free of dust, oil, etc.

b. Scratch the entire surface with a fine suede brush with uniform, firm strokes so that each region of the surface is abraded 2-3 times. The strokes should be in two directions perpendicular to each other.

c. Apply finely powdered graphite (Dixon's Air-spun, Type 200-10) to a soft cloth fastened over a sponge rubber pad fixed on a wooden block of convenient size.

d. Rub the abraded surface with this graphited cloth, applying more graphite to the cloth as needed to get a uniform covering of the surface. Rub in all directions and with sufficient pressure to make the graphite adhere to the surface.

e. Rub the entire surface with a clean cloth on a rubber pad, applying considerable pressure or with a soft buffing wheel and remove the darker spots where the coating is not uniform.

f. Measure the surface resistance systematically over the entire coated surface. (In every 3" x 3" square on large surfaces). Make additional tests in any area where the resistance is in the range of 1-10 megohms.

g. Rub on or rub off sufficient graphite to bring the resistance to the lower limit of the 1-10 megohm/ square range. (The protective coating will increase the resistance by a few megohms).

h. Check the light transmittance on any areas where the deposit of graphite seems too heavy. Rub off the excess graphite to bring the transmittance to 85% or above and recheck the surface resistance to maintain the specified values.

2. Preparation of Protective Overcoating

The recommended copolymer (ES-1) for overcoating is prepared as follows:

a. Mix 0.2 moles of methacrylic acid and 1 mole of methyl methacrylate monomer in 2 moles of cyclohexanone with 0.2% of benzoyl peroxide as the catalyst.

b. Reflux the mixture at 137-8°C. for one hour.

c. Cool in an ice-water bath.

The recommended formulation (ES-4) for spraying on the graphited surface consists of:

Copolymer ES-1	1 part
Butyl Acetate	2 parts
Methyl Ethyl Ketone	1 part

3. Application of Overcoating

a. Spray the material to be coated with a uniform coating of formulation ES-4.

b. Test the surface resistance and light transmittance systematically, as above. If these values are above the specified tolerances, the coatings must be removed and reapplied.

4. Used Canopies or Marred Surfaces

For canopies previously marred or scratched in service, the above procedure is modified as to the initial abrading operation.

a. Clean the surface thoroughly with an abrasive polish (such as Du Pont No. 7 Polishing Compound).

b. Remove oily or waxy residues with a cleaning solution (such as Du Pont No. 7 Polish and Cleaner).

c. On canopies, scratch the 2-inch band around the edge with crocus cloth. Otherwise no further abrasion is required.

d. Anneal the specimens or canopies by retention at 70-100°C. for one hour and cooling at 15°C. per hour to 50°C.

e. Apply the graphite and protective overcoating as above.